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RESEARCH ON COTTONSEED AND COTTONSEED PRODUCTS AT THE SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

A BIBLIOGRAPHY

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SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

For purposes of research by the U. S. Department of Agriculture on utilization of agricultural crops, the country has been divided into four regions. Each region is served by a Utilization Research and Development Division of the Agricultural Research Service. North Carolina, Oklahoma, Puerto Rico, South Carolina, Tennessee, and Texas. Headquarters for the Division are located at:

Southern Regional Research Laboratory
1100 Robert E. Lee Boulevard
New Orleans, La.

This laboratory conducts research on utilization of cotton, cottonseed, rice, peanuts and other oilseeds, and sweetpotatoes, and on fundamental chemistry and process engineering and development applicable to utilization of these crops. Four field stations in the Division are located near sources of supply of the raw materials being studied, including citrus and other subtropical fruits, pine gum and its derivatives, and cucumbers and miscellaneous southern-grown vegetables. These field stations are:

Naval Stores Laboratory, Olustee, Fla.
U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Fla.
U. S. Food Fermentation Laboratory, Raleigh, N. C.
U. S. Food Crops Utilization Research Laboratory, Weslaco, Tex.

For information on any of the lines of research being conducted in the Southern Utilization Research and Development Division, you are invited to write or visit the Southern Regional Research Laboratory, or the field station immediately concerned with the product in which you are interested.

RESEARCH ON COTTONSEED AND COTTONSEED PRODUCTS AT THE SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

A Bibliography of Publications, With Abstracts

By Marie A. Jones

Southern Utilization Research and Development Division
Agricultural Research Service

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QUARTER-CENTURY OF UTILIZATION RESEARCH AIDS COTTONSEED INDUSTRY

Research on cottonseed and cottonseed products has been an important part of the Southern Utilization Research and Development Division (SURDD) program since its beginning as the Southern Regional Laboratory, which was opened in 1941 and later became the Southern Regional Research Laboratory. Results of these efforts have been reported in the nearly 1,000 technical papers and patents listed here.

Only a little more than a century ago cottonseed was generally regarded as a waste product of the cotton industry, to be disposed of at some trouble and expense. So radically has this situation changed, however, that today the nine to 11 billion pounds of cottonseed produced annually in the United States return approximately $250 million to the grower. After processing, the products attain a value of about $1.5 billion at the consumer level.

Although production of cottonseed is generally considered incidental to the production of lint cotton, the seed is an item in the livelihood of the approximately 2,300,000 persons living on farms on which cotton is grown. Some 418,000 workers not living on cotton farms are employed there at the peak of the season. In addition to these, the cotton gins employ some 51,000 workers, and cottonseed oil mills another 12,000 during operating periods. To these numbers can be added 3,000 more employed in plants manufacturing shortening and cooking oils.

Research has been largely responsible for converting this erstwhile waste product into one of great value, both from the economic standpoint and as a source of products to meet the needs of humanity, directly or indirectly. Many individuals, industrial firms, independent research organizations, universities, and state and Federal agencies have participated in the research responsible for these advances, and none more vigorously than the cottonseed industry itself through the National Cottonseed Products Association and other representative organizations.

When the Southern Regional Research Laboratory entered the picture, cottonseed processors were seeking to increase the efficiency of their operations and to improve the quality of the oil, which was their most important product. They also hoped to increase the utilization of cottonseed meal through entrance into new markets. At that time the meal was used principally as a protein supplement for the feeding of cattle and other ruminants. Members of the cottonseed industry were convinced that through better processing methods meal could be produced that would be suitable for feeding to poultry, swine, and other nonruminants, thus providing access to these rapidly expanding markets.

With these goals in view, the SURDD undertook studies of the effects of time, temperature, and relative humidity during storage on the properties of the seed and the quality of the ultimate products. Optimum conditions of storage were recommended. Chemical treatments to retard deterioration were also investigated. Information concerning the influence of storage conditions on the formation of free fatty acids and off-color in oils and, particularly, concerning the growth of micro-organisms on the seed, valuable at that time, may be even more valuable today.

Research on the adaptability of solvent extraction to cottonseed processing aided and encouraged the industry to convert to this more efficient method of separating oil and meal. A new process, filtration-extraction, was also developed and is now being used successfully in at least two mills in this country and several in foreign countries.

The SURDD investigated other extraction methods, including hydraulic and screwpressing, to determine the effects of variations in cooking, flaking, crisping, moisture levels, and other processing conditions on the yield and quality of both oil and meal. From this information researchers were able to recommend methods conducive to the production of meal with high nutritive value and low gossypol content, suitable for feeding to growing chicks and swine. Such information has been largely responsible for opening these new markets to cottonseed meals. Cooperative feeding tests have demonstrated that properly processed cottonseed meals are equal in nutritive value to other products commonly used as sources of protein concentrate in rations for these animals.

Gossypol, the principal pigment in cottonseed, is generally blamed for adverse effects of cottonseed meal in the diet of nonruminants, and studies of gossypol were undertaken early in

the research of the identified several derivatives and related pigments. They also developed a flotation method of separating the pigment glands from the remainder of the seed. Methods of determining gossypol quantitatively were developed and levels of toxity fixed.

Because of the effects of gossypol and other complicating factors on the growth response of nonruminants fed cottonseed meal, a simple, reliable method of chemical analysis for measuring nutritive value assumes great importance, and much time and effort has been directed toward the development of such a method. The method of analysis for available lysine developed at the SURDD is widely accepted as the best method yet found. It agrees well with results of biological assay. However, simpler, more accurate methods are still being sought.

An economically feasible method of producing a cottonseed meal with high nutritive value and low gossypol content, together with high yields of good quality oil, has been sought by everyone engaged in processing research. A few years ago investigators at the SURDD announced the mixed-solvent extraction process, in which a mixture of acetone, hexane, and water (AHW) is employed to extract the oil. Engineering studies have been made to scale the process up to pilot-plant operations. Meals prepared by mixed-solvent extraction meet requirements for an extremely low content of both free and total gossypol. Heat damage to protein is avoided, so that nutritive values are preserved.

AHW meals show up extremely well in biological and clinical tests. When fed to young children as a source of protein, these meals produce growth responses similar to those of milk protein. For this reason the process is receiving the serious consideration of UNICEF officials and others interested in cottonseed as a source of protein for human nutrition in cotton-producing countries where supplies of protein are presently inadequate. In rations for swine and poultry, the AHW meals compare well with protein supplements from other sources, without the evidences of gossypol toxicity, egg discoloration, and other adverse conditions that sometimes result from the ingestion of ordinary cottonseed products.

The AHW process reduces to a very low level the Halphen acid response of the meal indicative of the cyclopropene fatty acids, depending on the amount of residual lipids. Of potentially greater value, in recent studies of the aflatoxins AHW extraction hes been found to remove these toxic materials from various oilseed materials. Its use has been suggested for preparing peanut and other meals for human consumption in areas where the aflatoxins are a problem.

Research on cottonseed and its products today is on a much more sophisticated plane than it was when the Southern Regional Research Laboratory first entered the field. The mycotoxins and the cyclopropenes are now of primary interest, particularly the mycotoxins. Fortunately, neither the mycotoxins nor the cyclopropenes have as yet become real problems to the cottonseed industry; as a speaker observed at a recent conference here on cottonseed research, "We are in the fortunate position of seeking solutions to these problems before they arise."

The Halphen acid response has been recognized as a characteristic of cottonseed oil for some 50 years; it is only quite recently, however, that the cyclopropene fatty acids have been identified as the cause of this response. Malvalic acid is the cyclopropenoid present in cottonseed in the highest proportion, but some sterculic acid is also present. These compounds are held to be largely responsible for the development during storage of a pink color in the whites of eggs produced by hens fed cottonseed meal, as well as for changes in the pH of such eggs and for other physiological effects. The role of gossypol in producing discoloration of the yolks in such eggs is well known. The effects of the cyclopropenoids and of various forms of gossypol on egg color are being investigated further.

The first step in coping with the problem of the cyclopropenes was to find reliable, sensitive methods by shich their presence could be determined quantitatively. Several such methods have been developed. Researchers are now endeavoring to concentrate, isolate, and study the cyclopropene acids from cottonseed oil.

Three effective methods for removing or inactivating the cyclopropenes have been developed at the SURDD. The one announced most recently is the treatment of cottonseed oils with certain acids, including cottonseed oil acids, or capric, oxalic, or acetic acids, in connection with the deodorization process. Another method selective hydrogenation at low temperatures preferentially saturates the cyclopropene groups. The third method is the use of activated alumina treated with sulfurous acid, mentioned later in connection with the bleaching of off-colored oils.

The cottonseed industry, along with other segments of the food and feed industries, has become concerned about the mycotoxins, poisonous materials produced by molds. Concern about these substances was aroused first by the discovery that the outbreak of "Turkey X" disease in the United Kingdom had been caused by the presence of aflatoxin, produced by the mold, Aspergillus flavus, in peanut meal. An intensive program of research by agencies of the U. S. Department of Agriculture, including the SURDD, was focused on peanuts first, but has been extended to take in cottonseed and other oilseeds.

It has been demonstrated at the SURDD that A. flavus can grow on cottonseed and cottonseed meal and produce toxin. Investigators here found early in their studies that assay methods used for peanuts were not applicable to cottonseed; so they developed an assay method especially for the latter. The procedure is sensitive to about 1 part per billion in cottonseed meats and 4 parts per billion in cottonseed meal. It appears to be appoicable to many other agricultural commodities, and several laboratories are already using it, not only for cottonseed but also for a number of other products. Instructions for the procedure have been distributed widely.

Another aid for analysis is a working standard prepared at the SURDD comprising a chloroform solution of all four known aflatoxins. Sealed ampoules containing 5 ml. aliquots have been distributed to various laboratories in response to requests. As previously mentioned, the AHW extraction process is effective in removing aflatoxin from oilseed meals, thus promising a ready solution should these substances become a serious problem. Early studies on the effects of storage conditions, however, afford guidelines for the prevention of mold growth and the contamination of cottonseed with mycotoxins.

If it appears that more research effort at the SURDD has been expended on cottonseed meal than upon the oil, it is because oil processing was much further advanced at the time the Division came into being and because the problems are less complex. A perusal of the abstracts pertaining to oil research will reveal, however, that much work has been done in this ares and that a great amount of information on the chemistry and processing of oil has been accumulated.

The greatest problem in oil production has been with color. Much of our early work on the storage and handling of cottonseed has a direct bearing on oil quality. Any adverse conditions of storage, such as relatively high temperatures and humidity, are also conducive to color fixation and the formation of free fatty acids in the oil.

Studies have been made of the effects of various conditions of preparation and processing on both yield and quality of the oil. Physical and chemical properties have been studied, and a mass of information has been developed on the behavior of the various fatty acids and their derivatives. New information has been provided on the hydrogenation and winterization of cottonseed oil.

Of the new products designed to increase the use of cottonseed oil, the acetoglycerides have been the most extensively commericalized. These products can be "tailored" to the consistency desired for a specific purpose. They range from oils to waxy fats suitable for food coatings and hard, brittle fats for use where this property is desirable. Some of these products maintain their texture over a wide range of temperatures from hot to cold. The acetoglycerides have good keeping quality, being much more resistant to rancidity than are ordinary fats. First used in cosmetics, the acetoglycerides are now widely used for food coatings.

Several other specialty fats have been made from cottonseed oil, among them some with properties closely resembling those of cocoa butter. The most promising of these is made by selective hydrogenation of stearin removed during solvent winterization of cottonseed oil. Plans are being made and equipment is being constructed for commercial trials of this product.

A number of plasticizers have also been prepared from long-chain fatty acids, and some of these plasticizers have shown up well in preliminary tests. The amide of stearic acid and bis(2-ethoxyethyl) amine is compatible as a plasticizer for vinyl chloride resins and other polymers. This amide also has premium properties of high efficiency and low volatility. The amide from oleic acid and dibutylamine is particularly attractive as an efficient plasticizer. Many of the amides have antimicrobial properties.

Utilization of cottonseed soapstock has been rendered more profitable through development of a process for making methyl esters directly from an acidulated soapstock. These ester products have been produced commercially for several years and marketed as a high-energy fat additive for poultry and livestock feed. The same process is applied to soapstock from corn and soybean oils.

Prospects for linters, another byproduct of cottonseed processing, are brightened by the development of an improved cotton batting through a cooperative research project initiated here a short time ago. This new type batting, Cotton Flote, produced by treatment with a mixture of resins and latices, has much greater resiliency and cohesion than does ordinary batting, and can be cut and molded in much the same way as the foams and similar products. Major automobile manufacturers are now using this batting in current lines of cars instead of foams, indicating the regain of a larger share of one of the most important cotton batting mardets. Furniture and bedding manufacturers are also using Cotton Flote in some of their top lines, and it is felt that the regain of these markets also is largely contingent on ability of processors to supply sufficient quantities of the new material.

The products and processes mentioned here represent some of the highlights of the accomplishments in cottonseed research at the SURDD over nearly 25 years, since the opening of the Southern Regional Research Laboratory. It is with pleasure that we present this abstract bibliography of publications reporting our research on cottonseed and its products as our contribution to the growth of one of the major industries based on a southern agricultural commodity.

We do not claim sole credit for these achievements; many workers and organizations have contributed to advances in cottonseed science and technoloty. We are particularly gratified by the close association we have enjoyed with the cottonseed industry as a whole. The cooperative spirit of its members, their appreciation of the value of research, and their eagerness to adopt new findings have contributed materially to the growth of the industry. Developments in the past decade indicate that the demand for cottonseed should continue to increase as its usefulness is extended into new areas. Products such as these from the vegetable proteins are being presented in new ans appetizing form to meet special nutritional needs. Improved conditions in many countries, especially those now entering periods of accelerated economic development, and the population explosion around the world should increase the demand for the oil and protein from cottonseed and make utilization research on these products even more rewarding in the future than in the past.

C. H. Fisher, Director
Southern Utilization Research
and Development Division

3322. CHEMICAL COMPOSITION STUDIES OF SEEDS OF THE GENUS GOSSYPIUM. Carter, F. L; Castillo, A. E.; Frampton, V. L.; and Kerr, T. Phytochemistry 5: 1103-12. 1966.

The amino acid patterns of the seed proteins and the fatty acid patterns of the seed oils were determined for 11 species of the genus Gossypium. A genus specific pattern was not found for either the amino or the fatty acids. The greatest difference among the amino acids was noted for lysine, which ranged from 4.12 g/16 g of meal nitrogen for G. hirsutum L. to 4.82 for G. klotzschianum var. davidsoni (Kellogg). A preliminary survey was made of the qualitative differences in the free amino acids in the ethyl alcohol extract of the defatted meals; there was considerable variation in the chromatographic patterns from the different species. Palmitic acid in the seed oils was observed to vary from 22 percent for G. arboreum L. to about 35 percent for G. aridum (Rose and Standley) Skovsted; oleic acid from about 16 for G. aridum to about 36 for G. armourianum Kearney; linoleic acid from about 35 for G. armourianum Kearney to about 55 for G. hirsutum L. Gossypol was noted to vary from 0 percent for the glandless variety of G. hirsutum L. to more than 9 percent for G. klotzschianum var. davidsoni (Kellogg). Statistical analyses of the data indicate that genes from the wild species should be of value in the production of commercial genotypes with improved proteins and oils.

3195. SEED PROTEINS. Altschul, A. M.; Yatsu, L. Y.; Ory, R. L.; and Engleman, E. M. Ann. Rev. Plant Physiol. 17: 113-36. 1966.

This is a review article dealing mainly with storage or reserve proteins in seeds. Other proteins that are present in small amounts, e.g., the toxin ricin, trypsin inhibitors, hemagglutinins, and allergens, are discussed.

3176. ONTOGENY OF ALEURONE GRAINS IN COTTON EMBRYO. Engleman, E. M. Am. J. Botany 53: 231-37. 1966.

Cotyledon mesophyll cells in maturing seeds of Gossypium hirsutum form aleurone grains by accumulation of protein and other materials in vacuoles. Globoids and unidentified electron-dense particles can be found in the matrix of amorphous protein. As revealed by electron microscopy, the vacuoles appear to communicate with cisternae of endoplasmic reticulum in early stages of development. This paper and many other reports in the literature cast doubt on the reported plastid nature of certain aleurone grains.

3169. MULTIPLE FORMS OF ASPARTATE OXOGLUTARATE TRANSAMINASE AND MALATE DEHYDROGENASE IN RESTING COTTONSEEDS. Fasella, P.; Bossa, F.; Turano, C.; and Rossi Fanelli, A. Enzymologia. Acta Biocatalytica 30: 198-205. 1966.

Aspartate oxoglutarate transaminase was purified from cottonseeds about 2700 fold. The preparation was analyzed by starch gel electrophoresis using a special technique to make evident enzyme activity on the gel. It has thus been possible to show that the preparation contains three protein bands provided with aspartate oxoglutarate transaminase activity; these bands display also malate dehydrogenase activity.

3168. PURIFICATION AND PARTIAL CHARACTERIZATION OF ALANINE OXOGLUTARATE TRANSAMINASE (AOT) FROM RESTING COTTONSEEDS. Turano, C.; Bossa, F.; Fasella, P.; and Rossi Fanelli, A. Enzymologia. Acta Biocatalytica 30: 185-97. 1966.

AOT was purified from cottonseeds about 5000 fold. The mechanism of action was analyzed by steady state kinetic studies. The results suggest that the reaction proceeds through the

successive formation of two enzyme-substrate binary complexes. The effect of some specific inhibitors and the pH dependence of some kinetic parameters were also studied.

3097. STUDIES ON COTTONSEED ENZYMES. I. PROTEOLYTIC AND GLUTAMIC DEHYDROGENASE ACTIVITIES IN COTTONSEED. Rossi Fanelli, A.; Cavallini, D.; Mondovi, B.; Wolf, A. M.; Scioscia-Santoro, A.; and Riva, F. Arch. Biochem. Biophys. 110: 85-90. 1965.

An acetone powder extract was prepared from delinted glandless cottonseed. A large production of various amino acids, α-ketoglutarate, and other keto acids were observed when the extract was incubated. The results obtained suggest that, in the cottonseed acetone powder extract, a proteolytic system is present, producing glutamate, which in turn is oxidized by glutamic dehydrogenase.

2938. CALORIMETRY FOR BIOCHEMICAL ANALYSIS. Evans, W. J.; and Carney, W. B. Anal. Biochem. 11: 449-59. 1965.

Thermopile elements of unique design have been incorporated in the construction of a Tian-Calvet calorimeter. The manner in which these sensing elements are made greatly facilitates building an instrument of this type. By use of a conventional dc amplifier and recorder, heat changes from 0.003 cal up to 1 cal, for both fast and slow reactions, can be measured.

2899. THE ULTRASTRUCTURE OF COTYLEDONARY TISSUE FROM Gossypium hirsutum L. SEEDS. Yatsu, L. Y. J. Cell Biol. 25: 193-99. 1965.

Quiescent cottonseeds stored in a dry, anaerobic situation for over a year were shown to contain cells with contents that are ultrastructurally similar to those of normal, fully hydrated plant cells. Plastids, mitochondria, and nuclei of the cells of cotyledon tissue in dry seeds possess normal-looking double membranes even under conditions of extreme desiccation. Previous reports indicate, on the basis of light microscopic work, that the cells of certain dry seeds do not possess nuclear membranes or mitochondria. The cells of the dry cottonseed do contain these structures, however. Dictyosomes have not been observed in the spongy parenchymal cells of the cotyledon tissue; it is suggested that they are concerned with translocation or utilization, or both, of material. The storage materials in the cells, protein and oil, are contained in vacuolar areas enclosed by a single membrane.

2853. INTRACELLULAR DISTRIBUTION OF SEED PROTEINS. IMPLICATIONS FOR FOOD SCIENCE. Altschul, A. M.; Dechary, J. M.; and Evans, W. J. Intern. Congr. Food Sci. & Technol., Proc., 1st, London, 1962. [Publ. 1964].

Seeds are the major source of protein for man and animals. Among these, the oilseeds represent a source of protein that has not realized its full potential. In the search for increased protein supplies for present and future world populations, increased attention will be given to more efficient use of proteins from oilseeds. This will require more sophisticated handling of the raw materials to put them in the best utilizable form. The proteins of peanuts were chosen for detailed study by DEAE cellulose chromatography and zone electrophoresis and by controlled rupture of the cells in such ways as to leave intact subcellular fractions containing the proteins. (Approximately 75 percent of the proteins in the peanut cotyledon exists in particles.) These are the less soluble proteins and generally have a lower nutritive value than the smaller proportion of proteins that are apparently free in the cytoplasm. Food science must take into account this segregation of the proteins and other seed contents in developing processes to allow maximum utilization of the seed proteins and permit a minimum of deleterious changes to take place during processing. Recognition of this compartmentalization adds another dimension to the opportunities of food science.

2814. RIBONUCLEOPROTEIN PARTICLES FROM STORAGE TISSUE OF MATURE SEEDS. Phillips, Marshall. Biochem. Biophys. Acta 91: 350-51. 1964.

A method is presented for the isolation of ribosomes from the storage portion of mature Arachis hypogaea and Gossypium hirsutum seeds. The dry seeds were homogenized in cottonseed oil and separated into subcellular components by differential centrifugation. The ribosomes were isolated from the RNA-rich fraction. Characterization of the particles as to UV spectra, RNA-protein content, and sedimentation coefficients revealed them to be similar to ribosomes from other sources.

2808. LIPOLYSIS AND THE FREE FATTY ACID POOL IN SEEDLINGS. St. Angelo, A. J.; and Altschul, A. M. Plant Physiol. 39: 880-83. 1964.

Lipolytic activity at pH 4-5 was observed in macerates of tissues from castor beans, peanuts, and cottonseeds germinated 4, 7, and 3 days, respectively. P-chloromercuribenzoate inhibited lypolysis in the castor seedlings. The peanut exhibited two maxima of lipolytic activity as germination progressed: 7 and 13 days. The latter was attributed to molds. When lipolysis is minimized during maceration, the long-chain free fatty acid content in the different species tested was less than 0.35μ moles per seedling. This value began to increase in the peanut as that seedling germinated past the 8th day. It is concluded that the free fatty acid pool in germinating seeds is very small. In those instances where substantial amounts have been observed, this could be either an artifact of the conditions of maceration or due to mold growth.

2769. SEED PROTEINS. Altschul, A. M. Proc. Symp. Foods: Proteins and Their Reactions, Corvallis, Ore. 1963, Chap. 13, 295-313. Publ. 1964 .

The major globulins of seeds can be investigated by chromatography on DEAE cellulose and by gel electrophoresis, particularly on polyacrylamide gel, as well as by sedimentation. Purified protein fractions undergo association-dissociation reactions with change in pH and ionic strength, can be dissociated by urea and other reagents into fragments of molecular weight below 20,000, and show relatively less organization and masking of reactive groups compared to other globular proteins. Most major globulins are found in sub-cellular particles associated with storage carbohydrate, fat, and phosphorus. The location of these proteins within the cell and their characteristic molecular properties influence their reactions during processing of seeds and their behavior when isolated.

2768. A NEW CLASSIFICATION OF SEED PROTEINS: APPLICATION TO THE ALEURINS OF ARACHIS HYPOGAEA. Altschul, A. M.; Neucere, N. J.; Woodham, A. A.; and Dechary, J. M. Nature 203, 501-504. 1964.

An approach to classification of seed proteins is proposed that uses an element of uniqueness in seeds as the basis for classification rather than solubility. Since much of the protein contained in seeds exists in so-called aleurone grains, or protein bodies, the proposal is made that those proteins thus contained be named "aleurins." This approach to classification is illustrated by consideration of the soluble proteins of the cotyledon of Arachis hypogaea.

2722. ISOLATION OF A MONODISPERSE PROTEIN FRACTION FROM COTTONSEEDS. Rossi-Fanelli, A.; Antonini, E.; Brunori, M.; Bruzzesi, M. R.; Caputo, A; and Satriani, F. Biochem. Biophys. Res. Commun. 15: 110-15. 1964.

Aqueous extracts from seeds contain a number of different protein components that vary from seed to seed, relatively few of which have been isolated as pure proteins. A simple procedure is described that allows preparation from cottonseeds of a protein fraction that behaves as a homogeneous monodisperse material. The name Acalin A is proposed for this protein, which appears to be one of the

major protein components of cottonseed. It shows a marked dependence of solubility on temperature and this property forms the basis of the isolation procedure. In this respect and in its other molecular properties it is similar to other "cold precipitable" globulins of different seeds.

2635. LIPID-PROTEIN PARTICLES: ISOLATION FROM SEEDS OF GOSSYPIUM HIRSUTUM. Yatsu, Lawrence; and Altschul, A.M. Science 142: 1062-64. 1963.

Subcellular particles were isolated from cottonseed by treating the cell contents with tannic acid. The two classes of particles, high in protein content and containing approximately 28 and 44 percent lipids, are thought to be the site of oil storage and lipid synthesis.

2107. CHARACTERIZATION OF OILS FROM LOW-GLAND AND GLANDLESS COTTONSEED. Thaung, U.K.; Gros, Audrey; and Feuge, R.O. J. Am. Oil Chemists' Soc. 38: 220-24. 1961.

New varieties of cotton that contain few or no gossypol pigment glands are being developed. In overall characteristics, oils from low-gland and glandless seed were indistinguishable from oil from ordinary glanded seed. Iodine values, contents of unsaponfiables, cloud- and pour-points, response to the Halphen test, and similar characteristics resembled those of commercial cottonseed oil. No differences were found in behavior during winterization. Determination of the component fatty acids by gas chromatography showed the fatty acid composition of the oils to be typical. Determination of the positions of the double bonds in the unsaturated acyl groups showed no differences between the oils from glanded, low-gland, and glandless seed. Ultraviolet, visible, and infrared spectra of the oils revealed no differences other than the presence of gossypol in the crude oil from the glanded seed. The infrared spectra of the unsaponifiable fractions showed some differences, which are not believed to be important.

2089. A COMPARISON OF CHEMICAL PROPERTIES OF SEEDS OF GOSSYPIUM SPECIES. Frampton, V.L.; Pons, W.A., Jr.,; and Kerr, Thomas. Econ. Botany 14: 197-99. 1960.

Marked differences were observed in the gossypol contents of the kernels of the species of Gossypium studied. The values ranged from 0.12 to 6.64 percent. No such differences were found in the lysine content of the protein expressed as grams per 16 grams of nitrogen.

1305. RELATIONS BETWEEN OIL, NITROGEN, AND GOSSYPOL IN COTTONSEED KERNELS. Stansbury, M.F.; Pons, W.A., Jr.; and Den Hartog, G.T. J. Am. Oil Chemists' Soc. 33: 282-86. 1956.

A study was made of the relations between oil, nitrogen, and gossypol contents of cottonseed kernels from the seed of eight commercial varieties of cotton grown at 13 locations during 3 years. Both environment and variety of seed had a highly significant influence on each constituent, whether expressed as percentage of the kernel or as weight of constituent per 100 kernels. On the basis of percent constituent in the kernel, each variety showed a significant positive correlation between oil and gossypol and significant negative correlations between oil and nitrogen and between gossypol and nitrogen.

1302. EFFECT OF MALEIC HYDRAZIDE APPLIED TO THE COTTON PLANT ON THE DEVELOPMENT OF FREE FATTY ACIDS IN THE SEED. Lambou, M.G.; Parker, N.S.; and Carns, H.R. J. Am. Oil Chemists' Soc. 33: 199-202. 1956.

The available evidence suggests that maleic hydrazide applied to cotton plants may have contributed to inhibition of the formation of free fatty acids in the seed during field exposure and the presampling period; the proliferation of mold on the seed at high relative humidities during storage; to reduction of rate of formation of free fatty acids. The seed appeared to be protected for approximately 16, 20, and 20 to 28 weeks at 100 percent, 91 percent, and 75 percent RH, respectively. The evidence seems to favor only slightly the higher concentration of treatment. However, its application prior to or at defoliation seemed to have no measurable effect on the development of free fatty acids.

1229. SIGNIFICANCE OF NEWER LABORATORY METHODS FOR ANALYSIS OF COTTONSEED PRODUCTS. Hopper, T. H. Oil Mill Gaz. 60(3): 28-29. 1955.

The long use of laboratory methods for the control of processing and the evaluation of products by the cottonseed industry is cited. The use of new methods for analysis is discussed briefly. Those mentioned include free and total gossypol, nitrogen (protein) solubility, and an improved method for determining residual lint.

909. AN ANALYTICAL SYSTEM FOR DETERMINING PHOSPHORUS COMPOUNDS IN PLANT MATERIALS. Pons, W. A., Jr.; Stansbury, M. F.; and Hoffpauir, C. L. J. Assoc. Offic. Agr. Chem. 36: 492-504. 1953.

Analytical procedures for the determination of total, inorganic, acid-soluble, phosphatide, and phytin phosphorus are described. Two colorimetric phosphorus methods used in the final evaluation of these different types of phosphorus compounds are given in some detail. Phosphorus present as nucleic acids or nucleotides and as carbohydrate esters may be calculated from the analytical data for the other types of phosphorus. Experimental evidence supports the choice of these phosphorus methods and their validity when applied to cottonseed, and the procedures should be equally suitable for the analysis of other oilseeds and plant materials.

886. INFLUENCE OF VARIETY AND ENVIRONMENT ON THE IODINE VALUE OF COTTONSEED OIL. Stansbury, M. F.; Hoffpauir, C. L.; and Hopper, T. H. J. Am. Oil Chem. Soc. 30: 120-23. 1953.

Data are reported on the variation of the iodine value of oil from seed of eight varieties of cotton grown at 13 locations during 3 years. Analysis of variance showed the influence of variety and of station-years to be highly significant statistically. Iodine value was negatively correlated with temperatures. Simple correlations for the relations between iodine value of the oils from seed of individual varieties and mean temperatures during two periods of boll and seed development were negative and highly significant. For the maturation period (35 days before boll opening) and the combined periods for squaring, fiber elongation, and maturation (73 days before boll opening) average reductions in iodine value per ° F. increase in temperature were 0.760 and 1.172 units, respectively.

870. PHOSPHORUS COMPOUNDS IN COTTONSEED KERNELS. INFLUENCE OF VARIETY OF COTTONSEED AND ENVIRONMENT. Stansbury, M. F.; Pons, W. A., Jr.; and Hoffpauir, C. L. J. Agr. Food Chem. 1: 75-78. 1953.

Information about the types and amounts of phosphorus compounds present in cottonseed is basic to any comprehensive consideration of the nutritive value of cottonseed products. Data are reported for seven types of phosphorus compounds in 58 samples of cottonseed kernels selected so that effects of varietal and environmental factors could be indicated. The influence of environment on the values for total, acid-soluble, phosphatide, inorganic, and phytin phosphorus, calculated to either a moisture-free or moisture-and-oil-free basis, was highly significant statistically as compared to variety. A significant correlation coefficient (+0.989) was obtained for the relation between total phosphorus and phytin phosphorus contents of the moisture-free kernels. The regression equation for this relation should prove useful for prediction purposes.

869. 2,3,5-TRIPHENYLTETRAZOLIUM CHLORIDE AS A RAPID INDICATOR OF VIABILITY IN COTTONSEED. Lambou, M. G. Science 117: 690-93. 1953.

A modification of Porter's method (Porter, R. H., Durrell, M., and Romm, H. J. Plant Physiol. 22: 149, 1947) of applying the tetrazolium chloride test to longitudinal sections of the whole cottonseed was applied to untreated seed and to seed that had been heat-treated or exposed to high-energy cathode rays and stored in a sealed container for several weeks.

Results were compared with those from the standard germination test on seed drawn from the same lots. Data indicated the possibility of using this strain by the modified procedure for rapidly detecting the germinating capacity of cottonseed. Modifications consisted of using a stronger solution of the stain than that used by Porter and removing the lint and coat from the kernel, since Porter had observed that these did not react with the dye. "Hard" seed (seed that did not imbibe water) were excluded because Porter found that they did not react.

844. APPLICATION OF LINE-WIDTH METHOD OF SPECTROGRAM EVALUATION TO SPECTROCHEMICAL ANALYSIS OF PLANT PRODUCTS. O'Connor, R. T.; and Heinzelman, D. C. Anal. Chem. 24: 1667-69. 1952.

A method generally applicable for the ready analysis of plant products has been devised and evaluated. It involves the preparation of ashes sufficiently buffered to permit the evaluation of metallic cations in most plant products from a single set of working curves. It uses an automatic recording microphotometer and measures the line width directly from the profiles on the recorder strip. This modification has simplified and increased the accuracy of this measurement. With its use the line-width method of measurement is satisfactory in reproducibility and permits measurements over a wide range of concentrations.

802. THE MEASUREMENT OF OXIDATION-REDUCTION POTENTIALS OF COTTONSEED SLURRIES. Condon, M. Z.; Andrews, F. M.; and Altschul, A. M. Plant Physiol. 27: 500-506. 1952.

In the method described, the importance of exercising care in the construction and cleaning of the platinum electrodes and the necessity of preparing the slurry under purified nitrogen are emphasized. Platinum electrodes, sensitive for approximately 20 experiments with resting seed, are "poisoned" after only one experiment with 5 day cotton seedlings. The potential-time patterns obtained with cottonseed slurries vary with chemical treatment and length of time of germination as well as with the age of the seed.

720. EFFECTS OF HIGH-VOLTAGE CATHODE-RAY IRRADIATION OF COTTONSEED. Lambou, M. G.; Mayne, R. Y.; Proctor, B. E.; and Goldblith, S. A. Science 115: 269-71. 1952.

Cottonseed has been irradiated with high-voltage cathode rays at dosages of 500,000 to 3,000,000 r. e. p. At 1 to 2 million r. e. p. the internal and external microflora were destroyed. Concomitantly, however, a reduction in germination and an inhibition of growth of the seedlings occurred proportional to the dosage of the rays. It is suggested that one or more enzymes associated with the normal phenomena of germination and growth have been inactivated.

653. TRACE METALS IN COTTONSEED KERNELS. Heinzelman, D. C.; and O'Connor, R. T. J. Am. Oil Chemists' Soc. 28: 373-74. 1951.

Spectrochemical analysis of cottonseed kernels of eight varieties of cotton grown at one location and one variety grown at 13 locations showed no typical differences in contents of copper, iron, and manganese associated with variety and location of growth. All samples contained traces of boron and zinc. They were not found to contain traces of aluminum, chromium, nickel, and tin.

495. GERMINATION AND FREE FATTY ACIDS IN SEED STOCK LOTS OF COTTONSEED. Hoffpauir, C. L.; Poe, S. E.; Wiles, L. J.; and Hicks, Martha. J. Am. Oil Chemists' Soc. 27: 347-48. (Also The Cotton Gin and Oil Mill Press 51(22): 30-31; The Cotton Digest 28(7): 28; Oil Mill Gaz. 55(5): 13-14.) 1950.

Examination of 254 samples of cottonseed, representing 16 varieties, indicated that the percentage of free fatty acids in the extracted oil may be used as an index for selecting lots to be reserved for seeding purposes. The percentage of germination decreased, on the basis of averages, with increasing free fatty acid content. The probability that a given lot of seed will exceed a specified minimum germination value decreases markedly as the free fatty acid value increases. The data allow such probabilities to be estimated from the free fatty acid values and suggest that cottonseed reserved for seeding, insofar as practical, have a low free fatty acid content, less than 0.75 percent in the oil.

426. SURVEY OF THE CHEMICAL COMPOSITION OF COTTON FIBERS, COTTONSEED, PEANUTS, AND SWEETPOTATOES. A LITERATURE REVIEW. Guthrie, J. D.; Hoffpauir, C. L.; Stansbury, M. F.; and Reeves, W. A. U. S. Dept. Agr. Bur. Agr. Ind. Chem. AIC-61 (Rev.): 116 pp. (processed). 1949.

A revision of a similar review published in 1944, this survey has a twofold purpose: to present available information about the composition of cotton fibers, cottonseed, peanuts, and sweetpotatoes in a convenient and useful form, and to show where knowledge of the composition of these commodities is inadequate or lacking. An attempt has been made to include information on all substances reported to be present in the commodities except where later literature has rather definitely shown the substance to be absent or incorrectly identified. References include 85 on cotton fibers, 119 on cottonseed, 134 on peanuts, and 94 on sweetpotatoes.

348. CATALASE AND PEROXIDASE IN GERMINATING COTTONSEED. Altschul, A. M.; Karon, M. L.; and Kyame, Lillian. Arch. Biochem. 18: 161-70. 1948.

To obtain information concerning the role of catalase and peroxidase in the production of energy, and especially to aid in developing methods for inhibiting their activity in cottonseed, these enzymes were investigated during the early period of germination. The amount of extractable catalase decreased and that of peroxidase increased: in one experiment, the amount of extractable catalase went down to 18 percent of its value in resting seed, whereas the extractable peroxidase rose 21-fold in the course of a 5 day period of germination. The decrease in the amount of extractable catalase during germination was observed to be much greater than the decrease in total catalase. It is suggested that one of the functions of catalase is to regulate, in conjunction with peroxidase, the amount of free energy of oxidation available for biological work.

318. NOTE ON THE HYGROSCOPIC EQUILIBRIUM OF COTTONSEED AND COTTONSEED PRODUCTS. Karon, M. L.; and Adams, M. E. J. Am. Oil Chemists' Soc. 25: 21-2. 1948.

These investigations are continued from an earlier study published in 1947 (J. Am. Oil Chemists' Soc. 24: 56-8. Item No. 255 in this list). The previous tests were made on whole cottonseed, cottonseed kernels, and undelintered hulls over a relative humidity range of 31 to 93 percent. The present article describes similar experiments over a moisture range of 11 to 93 percent on whole seed, delintered seed, kernels, linters, and lint-free hulls. Results, summarized in a graph and table show the equilibrium moisture content of dlintered seed to be slightly higher than that of whole seed. Linters had the lowest equilibrium moisture content of the various cottonseed products tested.

317. MISCELLANEOUS CONSTITUENTS. Dollear, F. G.; and Markley, K. S. Cottonseed and Cottonseed Products, Their Chemistry and Technology. Ed. A. E. Bailey, Interscience Publ., N. Y. 1948: 466-93.

The authors divide cottonseed into three main parts--kernels, linters, and hulls. Since the oil, protein and pigments of the kernels are covered elsewhere in the publication, only the

carbohydrates, phosphorus-containing compounds, mineral constituents, nitrogenous constituents other than protein, vitamins, and various minor components of this portion of the seed are included in this chapter. The discussion of linters devotes a section each to cellulose, pectic substance, mineral constituents, nitrogenous constituents, wax and resins, coloring matter, and water-soluble constituents. The analysis of hulls is broken down under hemicelluloses, mineral constituents, lignin, and tannins. It is pointed out that although considerable data are available concerning the miscellaneous constituents of cottonseed, many merit more intensive and detailed investigations, especially by application of the more recently developed tools and techniques. In addition, it is quite probable that other constituents are present in the seed.

313. BIOLOGICAL PROCESSES OF THE COTTONSEED. Altschul, A. M. Cottonseed and Cottonseed Products, Their Chemistry and Technology. Ed. A. E. Bailey, Interscience Publ., N. Y. 1948: 157-212.

Biological activity in cottonseed is discussed in terms of specific chemical and physical reactions and also with respect to its economic significance. The general properties of viable seeds and the biochemical methods used for studying them are outlined. Hygroscopic equilibrium, germination, heating, respiration, and lipolysis are discussed in detail with respect to methods of measurement and control. A section of the chapter is devoted to deterioration of seeds both in the field and in storage. Suggestions are made for minimizing or preventing this deterioration.

285. GERMINATION AND FREE FATTY ACID IN INDIVIDUAL COTTONSEEDS. Hoffpauir, C. L.; Petty, D. H.; and Guthrie, J. D. Science 106: 344-45. 1947.

The free fatty acid content of the nongerm half of individual cottonseed kernels was determined by a microtechnique. The germ end of the kernel was determined by a microtechnique. The germ end of the kernel was germinated, and viability was correlated with free fatty acid content. Data on 369 individual cottonseeds examined showed that in seeds containing less than 1 or 2 percent free fatty acid, on weight of kernel, the odds are better than 20 to 1 that the seeds will germinate. All seeds containing more than 5 percent free fatty acid, on weight of kernel, were dead.

255. HYGROSCOPIC EQUILIBRIUM OF COTTONSEED. Karon, M. L. J. Am. Oil Chemists' Soc. 24: 56-58. 1947.

The hygroscopic equilibrium of cottonseed has been investigated over a range of 31 percent to 93 percent relative humidity. From 31 percent to 71 percent relative humidity, the moisture content increased linearly from 6.03 percent to 10.27 percent, and from 71 percent to 93 percent relative humidity, moisture content increased rapidly from 10.27 percent to 22.19 percent. When cottonseed was separated into meats and hulls, including linters, the hulls were found to contain more moisture than the meats. It is apparent that when stored cottonseed is aerated, consideration should be given to the effect of local atmospheric conditions. The relative humidity of the air used for aeration can affect the moisture content of the stored seed either favorably or adversely. Although it may temporarily reduce heating by conduction of the heat of respiration, it may increase the moisture content and thus stimulate further respiration and heating.

234. A MANOMETRIC METHOD FOR THE DETERMINATION OF PEROXIDASE ACTIVITY, APPLICATION TO AN INVESTIGATION OF THE PEROSIDASE OF GERMINATING COTTONSEED. Altschul, A. M.; and Karon, M. L. Arch. Biochem. 13: 161-73. 1947.

A manometric method for the determination of peroxidase activity is described. It consists of reacting hydrogen peroxide with a suitable reducing substrate in the presence of peroxidase

for 5 minutes, after which the remaining unreacted peroxide is decomposed by means of catalase, and the amount of oxygen released is measured manometrically. The method has been applied to investigation of peroxidase from cottonseed, and the order of the reaction and effects of substrate concentration and pH have been determined. The kinetics of the effect of initial hydrogen peroxide concentration on cottonseed peroxidase activity has been investigated, and evidence has been obtained to support the hypothesis that peroxidase forms both active and inactive complexes with hydrogen peroxide.

222. COMPARISON OF RESPIRATION, FREE FATTY ACID FORMATION, AND CHANGES IN THE SPECTRUM OF THE SEED OIL DURING THE STORAGE OF COTTONSEED. Kyame, Lillian; and Altschul, A.M. Plant Physiol. 21: 550-61. 1946.

Samples of cottonseed were analyzed for free fatty acid content, and the lipolysis rate constant was determined for each sample. The lipolysis constants were plotted as a function of moisture content. Those for the Delfos seed fell on a smooth curve and those for the Coker's and Oklahoma Triumph fell on a second smooth curve, which was lower than the Delfos curve. It was found that the lipolysis constant can be converted into a linear function of the moisture content of the seed. A variation of the spectrum of solvent-extracted cottonseed oils during storage was found. The highest moisture samples of Coker's and Oklahoma Triumph showed a common pattern, which was in turn different from that exhibited by Delfos. The pattern of lipolysis for immature seeds differed from the pattern for normal seeds in that the rate of formation of free fatty acids decreased with length of storage. The Delfos seeds exhibited more vigor both with respect to lipolysis and respiration than did the Coker's and Oklahoma Triumph seeds.

221. RESPIRATION OF COTTONSEED. Karon, M. L.; and Altschul, A.M. Plant Physiol. 21: 506-21. 1946.

Effects on the respiration of cottonseed of such variables as moisture content, seed variety, location of growth, and age were studied on samples of three varieties of cottonseed grown in three widely separate locations taken at different times during the harvesting season. Complete respiration patterns were obtained on 17 different lots with moisture contents ranging from 10 to 17 percent. The experimental data are tabulated and illustrated. The respiratory quotient of resting cottonseed was found to be unity. A quantity designated "average respiratory intensity" (RI), defined as the average rate of respiration of the seeds over a period exceeding 100 days, was proposed, and a means for its measurement was developed. The values for RI in any one series of seeds were shown to be smooth functions of moisture content. Mature Delfos seeds respired at a higher level than did the Coker's or Oklahoma Triumph seeds. Analysis of the published results of other investigators on flaxseed and sunflower seeds showed that the respiration rate of flaxseed was very close to that of Coker's and Oklahoma Triumph cottonseed and the respiration rate of sunflower seeds was similar to that of Delfos.

220. DETERMINATION OF MOISTURE IN COTTONSEED AND COTTONSEED PRODUCTS. Hoffpauir, C. L; and Petty, D. H. Oil & Soap 23: 285-8. 1946.

Losses in weight of whole, crimped, fumed and ground cottonseed, whole and ground cottonseed meats, and meal were studied in both vacuum and forced draft ovens at 101°C. and in forced draft ovens at 130°. The values obtained were compared with moisture values by the Karl Fischer method. The amount of moisture in a sample had very little influence on the time required to attain a constant dehydration level for the conditions studied. With whole cottonseed, 12 to 16 hours' drying at 101° in the forced draft oven was satisfactory. With crimped seed, 8 hours at 101° was required to attain an equivalent degree of dehydration. Values about 0.2 percent lower were obtained in the 5 hours specified in the official method. With fumed and ground cottonseed, whole or ground meats, and cottonseed meal, 5 hours at 101°

was required to reduce the rate of loss to a minimum. This time interval is longer than the time specified in the official method (2 hours for fumed and ground cottonseed and 3 hours for meal and meats). On heating cottonseed materials in forced draft ovens, larger losses in weight were obtained at 130° than at 101°. At 101° both the vacuum and the forced draft ovens reduce cottonseed materials to equivalent degrees of dehydration, the time required is different.

181. DETERMINATION OF INORGANIC PHOSPHORUS IN PLANT MATERIALS. Pons, W. A., Jr.; and Guthrie, J. D. Ind. Eng. Chem., Anal. Ed. 18: 184-86. 1946.

An improved procedure is described for determining inorganic phosphorus in plant materials. The method is unaffected by large amounts of protein or by the colored or turbid solutions and reducing agents present in the materials. The major steps involved in the method are extraction with 0.75 N trichloroacetic acid, followed by colorimetric evaluation of inorganic phosphorus by a modification of the Berenblum and Chain shakeout procedure. Separation of inorganic phosphorus from easily hydrolyzable organic phosphorus is obtained in the shakeout step. The final molybdenum blue complex is stable up to 19 hours after development. It has absorption maxima at #625 to #630 m. and 730 m., respectively. Tabulated data on the inorganic phosphorus content of various plant materials are given to illustrate the general applicability of the method.

176. UPTAKE OF HYDROCHLORIC ACID BY COTTONSEED AND COTTON FIBER DURING "FUMING" OF ANALYTICAL SAMPLES. Faust, A. R.; and Henson, F. A. Oil & Soap 23: 74. 1946.

Ten-g. samples of cottonseed with various percentages of linters, as well as 10-g. samples of cottonseed hulls and meats, raw and purified cotton fibers, and purified cotton linters, were burned in 2 g. portions in a Parr oxygen bomb. Chlorine was determined gravimetrically as silver chloride. The results are tabulated and show that cottonseed takes up 0.4 to 0.5 percent total chlorine and that all parts of the seed are capable of taking up hydrochloric acid. The effect of this is to lower the percentage of oil about 0.1 percent ant the total nitrogen by about 0.02 percent.

SEED - STORAGE AND HANDLING

2656. THE ARS 8' x 10' COTTONSEED CLEANING-BELT UNIT. INVESTIGATION OF ALTERNATE SURFACE TEXTURES. Gastrock, E. A.; Holzenthal, L. L; Gentry, W. T.; and Patton, E. L. Oil Mill Gaz. 68(7): 10-12. 1964.

The basic element for separating foreign matter from cottonseed in the ARS 8' x 10' Cottonseed Cleaning-Belt Unit is a resilient surface texture attached to the inclined horizontally moving belt. This texture traps the foreign matter from the "opened" feed and it is carried over the side to "rejects" while the seed is allowed to bounce and roll to "accepts" off the lower edge. Since only one belt surface texture has been used, improved operation was sought through use of an improved surface texture. This report gives results of static tests carried out with 3- by 8- ft. samples of 11 new resilient surface textures with the presently installed texture used as the basis of comparison. One texture was found which shows increased boll wall retention (26 percent) and better stem removal (9 percent) than the present texture but a slight (4-7 percent) increase in small seed retention. This increase in seed retention is not considered a handicap in use of the new texture.

2253. THE ARS 8' x 10' COTTONSEED CLEANING-BELT UNIT--IMPROVED OPERATION WITH BEATER-TYPE OPENER-FEEDER. Holzenthal, L. L.; Molaison, L. J.; Gentry, W. T.; Jr.; Gastrock, E. A.; and Patton, E. L. Oil Mill Gaz. 66(6): 5, 7-9. 1961.

The paper presents results obtained in single-pass experimental runs with the ARS 8' x 10' Cottonseed Cleaning-Belt Unit after replacement of its high capacity feeder-opener unit with a unit consisting of a modified conveyor with a prototype beater-opener unit. Improvement in cleaner performance was primarily due to "opener" performance, which distributed seed onto the moving belt as individual particles rather than in agglomerates. Operation of the cleaning-belt unit with the conveyor-feeder with beater-opener unit was satisfactory with input feed rates to about 30 tons per 24 hours. Seeds included cottonseed containing 0.7 to 2.4 percent total foreign matter and 8.5 to 12.0 percent linters, representing gin run seed, oil mill cleaned seed, and first-cut delintered seed. Results indicate possible use of this type cleaner at gins to reduce trash in seed ginned from rough-harvested cotton, and in oil mills as a supplemental cleaner either before or after first-cut delintering machines.

2190. EVALUATION OF A PROTOTYPE UNIT TO SEPARATE FOREIGN MATERIAL FROM COTTONSEED UTILIZING INCLINED PLANE MOVING SURFACES. Holzenthal, L. L.; Gastrock, E. A.; and Gentry, W. T., Jr. Oil Mill Gaz. 66(3): 9-14. 1961.

A radically new approach is described for the removal of foreign matter from cottonseed, particularly field trash, including stems, fragments of inner (shale) and outer boll wall, fine field trash, sand, dirt, and fibrous materials. Three pilot-scale arrangements were studied. Of these, the inclined horizontally moving one was found to be superior to the vertically moving or the circular-moving arrangements, based on input capacity feed rate, accepts to rejects ratio, cleaning efficiency, and ease of operation. Cleaning efficiency was referred to stems and shale, principally because oil mill operators agree they are most detrimental to second-cut linter quality. Designs were made for a larger unit using the best arrangement.

1873. PRELIMINARY RESULTS WITH THE ARS 8' x 10' COTTONSEED CLEANING-BELT UNIT. D'Aquin, E. L.; Holzenthal, L. L.; Gastrock, E. A.; and Gentry, W. T., Jr. Oil Mill Gaz. 64(1): 36-40. 1959.

A radically new type cottonseed cleaner called the ARS 8' x 10' Cottonseed Cleaning-Belt Unit is described, which is capable of removing stem and shale impurities from gin-run cottonseed to a greater extent than that obtained by conventional oil mill cleaner units and at comparable capacities. The principal element is a slow-speed, horizontally moving, rough-textured endless belt, inclined at an angle of about 30° . Data are presented to show that the machine can remove high percentages of the stems, shale and total foreign matter from cottonseed after it has been cleaned by a standard oil mill cleaner, and also after the mill-cleaned seed has been first-cut delintered. The machine produces two fractions. One is the cleaned seed product comprising about 93 to 98 percent of the total seed; the other is a concentrated trash fraction which contains the remaining seed. The latter fraction can be routed directly to the hulling. An alternate method that employs a simple, inexpensive, flaming technique, is described, by which practically 100 percent of the seed in the high-trash reject fraction can be recovered.

1858. APPLICATION OF THE ARS COTTONSEED CLEANING-BELT UNIT TO OIL MILL OPERATIONS. Gastrock, E. A.; Holzenthal, L. L.; D'Aquin, E. L.; Gentry, W. T.; Jr.; and Molaison, L. J. Oil Mill Gaz. 64(2): 55-58. 1959.

The ARS 8' x 10' Cottonseed Cleaning-Belt Unit has been described. Typical preliminary results show that at 25 tons per day, the stems content was reduced by 77 to 84 percent and the total foreign matter content was reduced by 44 to 56 percent, with seed representative of material immediately before and after cleaning by the usual oil mill pneumatic-mechanical cleaner machines. For seed representative of material passing through the first-cut delintering machines the respective reductions were 70 to 73 percent for stems and 37 percent for total foreign matter. Back-feeding about 15 percent of the output of the unit has some advantages. Installation of the cleaning belt unit at some point in the usual sequence of preparation operations at the oil mill (probably after the pneumatic-mechanical cleaner and before

first-cut delintering) should make possible the production of cleaner first-cut and second-cut linters. It will be necessary to conduct mill-scale tests of the cleaning-belt unit in order to determine its actual performance and capacity under mill conditions.

1857. ALIMENTADOR DE HOJUELAS DE MATERIALES OLEAGINOSOS (Flake-Feeding Device for Oleaginous Materials). Gardner, H. K.; D'Aquin, E. L.; Gastrock, E. A.; and Bourdette, V. R. La Hacienda 54(9): 38-39. 1959.

A feeding device was developed for use in feeding flaked oil-bearing materials to a pilot-plant size solvent extractor to provide a continuous uniform discharge of the material that can be varied over a 2.5 to 1 range; to form a positive seal plug to prevent the escape of solvent vapors at the point of entry of the material; and to cause a minimum breakage of the material into very small particles. The device operated satisfactorily with flakes from cottonseed, peanuts, okra seed, and rice bran. A desirable feature of the device is that it does not break up the material excessively. Another operating feature not incorporated in conventional screw-type feeders is elimination of material bridging above the feed screw. The unit is portable, can be economically constructed and operated, and is suitable for feeding a wide variety of materials other than oilseed flakes or meats. Scaling up to commercial size should be entirely feasible. The only alteration would be to extend the distributor screw through the side of the hopper so as to provide a material run-around for automatic feeding of the hopper.

1692. PROGRESS IN CLEANING COTTONSEED. Gastrock, E. A.; Holzenthal, L. L.; and D'Aquin, E. L. Oil Mill Gaz. 63(2): 40-41. 1958.

In research on cottonseed cleaning at SURDD traditional methods were avoided. Instead physical properties and differential responses of cottonseed and particles of foreign material not hitherto used commercially for separating contaminants from cottonseed have been investigated. Greatest attention has been directed to two new approaches: (1). Projection methods in which the cottonseed travels greater distances than the foreign material; and (2). rolling and tumbling methods in which the cottonseed roll or tumble faster than the foreign material. Recent results are described.

1382. RECENT WORK ON THE ARS DIFFERENTIATOR FOR CLEANING OF COTTONSEED. Gastrock, E. A.; Holzenthal, L. L.; Molaison, H. J., and Vix, H. L. E. Oil Mill Gaz. 61(3): 25-26. 1956.

A new principle in the cleaning and separation of cottonseed by slingshot projections as exemplified by the ARS Differentiator, developed at the Southern Utilization Research Branch, is discussed. The four basic elements of the ARS Differentiator, consisting of the feeder, opener, projector, and collector, are described, and their method of operation is explained.

1381. PRELIMINARY EXPERIMENTS ON CLEANING OF COTTONSEED WITH THE ARS DIFFERENTIATOR. Holzenthal, L. L.; D'Aquin, E. L.; Molaison, H. J.; Gentry, W. T., Jr.; and Vix, H. L. E. Oil Mill Gaz. 61(6): 19-24. 1956.

A pilot-plant machine called the ARS Differentiator is described for continuously cleaning cottonseed by casting it horizontally into air at a relatively high velocity. Principal element of the machine is the projector, which projects the seed a distance of about 100 feet. The Differentiator was operated experimentally in a series of eight single-projection runs in which the principal variables were evaluated. For the experiments reported, the results indicate that higher separation efficiency was obtained for low over high capacity or throughput rate, and for low over high clearance between the belts. Data are included to show that the seed that are projected farther are progressively higher in oil content, free fatty acids, and nitrogen. Application of the Differentiator to mill-scale operation is discussed.

1310. NEW APPROACH FOR THE CLEANING OF COTTONSEED. Holzenthal, L. L.; Gentry, W. T., Jr.; and Gastrock, E. A. Oil Mill Gaz. 60(11): 12-22. 1956.

A new approach is described for the cleaning of cottonseed wherein the seed and its contaminants are cast or projected horizontally into still air at high speed and subjected to the combined effects of air drag and gravitation. On a small scale, the spectrum-like floor pattern obtained shows possibilities of utilizing projection for removing high percentages of foreign matter from undelintered or partly delintered cottonseed. Results of three series of exploratory experiments are presented. The method also appears capable at the same time of separating the seed to an appreciable extent in accordance with their meat content, oil and nitrogen contents, oil quality, linters content, and length of linters.

1228. SEPARATION OF COTTONSEED BY PROJECTION--QUALITY OF FRACTIONS. Hozenthal, L. L.; Gentry, W. T., Jr.; Vix, H. L. E.; D'Aquin, E. L.; and Gastrock, E. A. Oil Mill Gaz. 60(4): 25-28. 1955.

Further experiments in the separation of cottonseed from intermixed foreign material have been carried out. A larger slingshot device than the one used in previous trials, gave higher velocity, and the tests are considered more accurate than previous ones because of the larger amounts of seed used. Samples contained foreign matter ranging in amount from 3 to 9 percent. Observations were made to determine: (1) The qualitative and quantitative distribution of the foreign matter in the various fractions; (2) the number of reprojections or recycles of the high-trash content fractions required to recover practically all of the seed as free of foreign matter as possible; and, (3) the differences in quality of seed in each fraction. Results were encouraging as to effectiveness of the method, not only for separation of the seed from foreign matter, but also for separation as to quality.

1092. PROGRESS REPORT ON CLEANING COTTONSEED. Holzenthal, L. L.; Gastrock, E. A.; Pollard, E. F.; Molaison, H. J; Gentry, W. T., Jr.; Martin, C. T.; and Crovetto, A. J. Oil Mill Gaz. 59(5): 14-16. 1954.

Two devices for cleaning cottonseed are described in this fourth progress report; one for removal of "sticks" or stems and the other for removal of all foreign material. Although exploratory data on the latter device are incomplete, indications are that the removal of substantially all trash from cottonseed may be achieved through this approach.

1013. COTTONSEED CONTENTS VARIATION: INFLUENCE OF VARIETY AND ENVIRONMENT ON OIL CONTENT OF COTTONSEED KERNELS. Stansbury, M. F.; Cucullu, A. F.; and Den Hartog, D. T. J. Agr. Food Chem. 2: 692-96. 1954.

Analysis of variance indicated that both variety and environment have a highly significant influence on the oil content. Oil contents of moisture-free cottonseed kernels from seed of eight commercial varieties of cotton grown at 13 locations during 3 years ranged from 26.8 to 43.4 percent. Rainfall influenced oil content significantly even with temperature constant; maximum temperature significantly influenced oil content with rainfall held constant. Although rainfall and maximum temperature during the maturation period are important causes of variation in the oil content, much of the environmental variation in oil content is also due to other factors not considered in the study. There was no statistically significant difference in the response of any of the varieties to variations in temperature and rainfall.

729. RESEARCH ON THE STORAGE OF COTTONSEED. Jensen, E. A.; Lambour, J. S.; Lambou, M. G.; and Altschul, A. M. Cotton Gin & Oil Mill Press 53(5): 68-69, 84-85. 1952.

Information not available at the time of the 1950 report and progress made in 1951 are reported. Also, accomplishments during the tenure of fellowship are summarized. In 1951

studies have been made of two methods of handling--heat and fungicides--as complementary agents to chemical treatment to inhibit deterioration of cottonseed during storage.

644. RESEARCH ON COTTONSEED STORAGE AND PROCESSING AT THE SOUTHERN REGIONAL RESEARCH LABORATORY. Altschul, A. M. Cotton Gin & Oil Mill Press 52(16): 24, 26, 28-30, 32. Cotton Digest 23(46): 6-7, 42-43, 46. 1951.

One objective of research on the storage of cottonseed has been the development of a principle of inhibition of biological changes in the stored seed by use of chemicals. Chemicals promising in the laboratory were tested in commercial storage. The production of heat and the rate of formation of free fatty acids were found to be dependent on the initial moisture and free fatty acids content. In a cooperative project with the Educational Service of the National Cottonseed Products Association, the Southern Laboratory is to provide nutrition investigators with cottonseed meals of known processing history. The investigators have found that experimentally made screw-pressed meals can be fed to hogs in a concentration up to 43 percent of the total diet and to chicks in a concentration up to 70 percent. Lower temperatures of cooking in the screw-press operation resulted in meals with higher nutritive value and in some that did not cause yolk discoloration in stored eggs even when fed at levels of 20 to 25 percent of the diet.

586. NEW IDEAS ON PROBLEMS OF STORAGE. Lambou, M. G.; Condon, M. Z.; and Altschul, A. M. Yearbook Agr., U. S. Dept. Agr. 1950-51: 551-57.

In the hot, humid climate which prevails during much of the year in the gulf coast states, the storage of any type of seeds is difficult. Natural drying in the field is not always possible, and methods of artificial drying have been devised, but drying alone does not prevent deterioration of the quality of the seed. A chemical treatment, which, combined with aeration, reduces biological activity in cottonseed to such a slow rate that there is a negligible rise in the content of free fatty acids and the color of the oil and no rise in the refining loss after 40 days' storage, is described in detail. Also described is a rapid method of producing deterioration of the seed in the laboratory for experiments to test the inhibitory activity of different chemicals. Laboratory and mill-scale experiments showed that the treatment is applicable also to flaxseed and rice. General application of chemical treatment will depend on the cost, toxicity, and effects of the materials on the finished products.

555. RESEARCH ON STORAGE OF COTTONSEED. Jensen, E. A.; Lambou, M. G.; and Altschul, A. M. Cotton Gin & Oil Mill Press 52(4): 17, 44-45. Oil Mill Gaz. 55(8): 55-59. Cotton Digest 23(20): 18-19. 1951.

The annual report of the Fellow of the National Cottonseed Products Association summarizes the year's progress in developing methods of improving the storage qualities of cottonseed. Mill-scale research conducted for several years in cooperation with industry was completed. Methods of handling were sought as complementary agents to chemical treatment to inhibit deterioration of stored seed. A method of heating cottonseed was developed and the effect of heat treatment was being investigated. The specific effect of fungicides on the spontaneous development of heat and on the formation of free fatty acids in stored cottonseed was being studied. An evaluation of some of the experiments could not be made since the work was not completed and was continuing.

554. THE STORAGE OF COTTONSEED. IX. BEHAVIOR OF COTTONSEED DURING STORAGE UNDER MILL CONDITIONS. Jensen, E. A.; Lambou, M. G.; Andrews, F. R.; Mayne, R. Y.; Karon, M. L.; Curet, M. E.; Wilcox, F. B.; Altschul, A. M., Newby, Wales; and Bollens, W. F. J. Am. Oil Chem. Soc. 28: 241-45. 1951.

With the cooperation of industry, an investigation was carried out from 1944 through 1950 on the behavior of cottonseed of various moisture contents during storage under mill conditions.

It was learned that both the production of heat and the rate at which free fatty acids are formed depend not only on the initial moisture content, but also on the initial free fatty acids content. A reduction in moisture content by sustained aeration did not control the rapid rise in free fatty acids content; the oil content appeared to decrease during storage; and the refining loss appeared to be proportional to the quantity of free fatty acids in the crude oil. The data are tabulated and illustrated by graphs.

451. RESEARCH ON THE STORAGE OF COTTONSEED. Jensen, E. A.; Lambou, M. G.; and Altschul, A. M. The Cotton Gin & Oil Mill Press 52(2): 11-12, 31, 34-35. 1950.

A number of mill-scale experiments have been conducted in cooperation with the cottonseed oil processing industry to test the soundness of conclusions based on laboratory experiments using chemical treatment and to obtain accurate information on the nature of the cottonseed storage problem. A mill-scale storage experiment is being conducted on freshly harvested cottonseed of a moisture content of about 18 percent and a free fatty acids content of about 3.7 percent. Only tentative conclusions can be drawn. Heating was prevented by chemical treatment and free fatty acid formation was delayed for approximately 52 days. Some of the data indicate the effect of storage on the amount of total oil available in the seed. In other experiments, cottonseed was collected at intervals over a period of 2 months and the biological properties of the seed were investigated. The results emphasized that cottonseed is not a constant biological material. An experiment was performed to determine the relationship between microorganisms growing on the seed and the seed enzymes themselves in the development of free fatty acids. Results indicate that there are at least two types of free fatty acid development in moist cottonseed: One type is rapid and seems to be associated with a rapid rise in growth of microorganisms; the other is gradual and takes place even in sterile seeds.

414. EFFECT OF TEMPERATURE ON THE CONTENT OF PIGMENTS OF STORED COTTONSEED. Castillon, L. E.; Hall, C. M.; O'Connor, R. T.; and Miller, C. B. J. Am. Oil Chemists' Soc. 26: 655-59. 1949.

Three purebred varieties of cottonseed, G. hirsutum, planted and grown under similar environmental conditions, were stored at 38°, 77°, and 85°F., after determination of their initial contents of lipids, nitrogen, moisture, gossypol, and gossypurpurin. The content of gossypurpurin in all the samples was found to increase in proportion to the temperature and length of storage, while the content of gossypol decreased. During storage another yellow-colored pigment developed, which could be separated from gossypol by alkaline extraction of the original chloroform extract of the stored seed. The alkali-extractable portion of the chloroform extract gave a red-colored antimony trichloride reaction product characteristic of gossypol. At least a fraction of the nonacidic pigment in the crude chloroform extracts obtained from stored cottonseed is thought to be diaminogossypol.

394. UNIFIED METHODS FOR THE ANALYSIS OF FATTY MATERIALS, INTERNATIONAL UNION OF CHEMISTRY: A TRANSLATION. Hoffpauir, C. L. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-225. 1949. (Processed)

This is a translation of the unified methods for the analysis of fatty materials of the International Chemical Union and constitutes the third report of the International Commission for the study of fatty materials published in 1948. It describes methods for the preparation and analysis of oleaginous seeds; methods for the analysis of fats and oils for moisture, impurities, ash, acidity, unsaponifiable matter, density, refractive index, preparation of insoluble fatty acids, determination of titer, saponification number, iodine number, hydroxyl number, oxidized acids, and polybromide number; and methods for the analysis of hard soaps for moisture, foreign substance insoluble in alcohol, total crude fatty acids, total alkali, total free alkali, rosin, and chlorides.

390. INHIBITORS OF HEATING AND DETERIORATION IN SEEDS. I. ETHYLENE CHLORHYDRIN AND RELATED COMPOUNDS. Condon, M. Z.; Lambou, M. G.; Vignes, J. L.; Loe, J. B.; and Altschul, A. M. Plant Physiol. 24(2): 241-54. 1949.

Ethylene chlorhydrin in concentrations of about 0.19 percent (dry-weight basis) successfully inhibited the formation of free fatty acids in moist flaxseed and cottonseed, while lower concentrations stimulated their formation. All concentrations of this particular chemical inhibited heating to some extent. A comparison of related inhibitors revealed that reduced concentrations do not necessarily have the same effect on both heating and free fatty acid formation, each inhibitor displaying a characteristic pattern of behavior in this respect. Three patterns of inhibitory behavior are described.

388. CHEMICAL TREATMENTS OF SEEDS TO PREVENT HEATING AND DETERIORATION DURING STORAGE. Altschul, A. M. Cotton Gin & Oil Mill Press 50(1): A2-10. 1949.

This article provides a summary of the present status of the research and developments on the problems involved in seed storage, and especially the more recent work being conducted at the Southern Regional Research Laboratory on cottonseed. Application of a rapid method for testing the ability of chemicals to inhibit free fatty acid formation in seed has resulted in the discovery of a number of widely different substances which can act as inhibitors in relatively low concentrations. It has been found that mixtures of two chemicals inhibit heating for much longer periods than does either one when used alone. Laboratory experiments show that a combination of mechanical aeration and chemical treatment is much more effective than either method alone.

355. STORAGE OF COTTONSEED AND PEANUTS UNDER CONDITIONS WHICH MINIMIZE SPECTROPHOTOMETRIC CHANGES IN THE EXTRACTED OIL. Pons, W. A., Jr.; Murray, M. D.; O'Connor, R. T.; and Guthrie, J. D. J. Am. Oil Chemists' Soc. 25: 308-13. 1948.

This work was undertaken to establish new indexes of deterioration and to verify previous recommendations for the storage of cottonseed and peanuts. The increase in cottonseed of red gossypol (a term used to designate substances exhibiting absorption maxima in chloroform at 528 and 565mμ), also increased absorption of the extracted oil in the region of 236mμ , and proved helpful in following changes in conditions during storage of the seed. Methods for applying both these indexes are described. In previous experiments cottonseed and peanuts with a moisture content of 8 percent or less showed no increase in free fatty acid content after a year's storage in closed containers at 1° or at -18° C. In the present studies cottonseed stored under the same conditions showed no appreciable change in red gossypol content, and little or no change in the nongossypol absorption of the extracted oil at 236mμ, the region of diene conjugation. When cottonseed was stored at room temperature, 27° C., both of these factors showed large increases. Oil extracted from peanuts stored for more than 4 years at room temperature showed a much greater absorption in the region of 227 to 234mμ than oil from peanuts stored at the lower temperatures.

354. EFFECT OF CHEMICAL TREATMENT PRIOR TO STORAGE ON VIABILITY AND GROWTH OF COTTONSEED. Lambou, M. G.; King, G. S.; and Condon, M. Z. Science 108: 563-4. 1948.

Previously reported investigations showed that a number of chemicals, including propylene glycol dipropionate and 4, 6-bischloromethyl xylene, prevent or minimize heating and deterioration of moist seeds in storage. This report covers studies to determine whether a mixture of these two compounds also could be used to prevent loss of viability. At intervals over a storage period of 6-1/2 months, germination tests and growth measurements were made on

treated and untreated cottonseed with a moisture content of 12 percent. Viability and growth of the treated seed were maintained at levels considerably higher than those of the untreated control. Analysis of the seed at the end of the 6-1/2 months confirmed previous findings on the effectiveness of these two chemicals in preventing the formation of free fatty acids during storage.

300. A RAPID METHOD FOR THE MEASUREMENT OF THE INHIBITION OF DETERIORATION IN INTACT SEEDS. Lambou, M. G.; Condon, M. Z.; Jensen, E. A.; Andrews, F. R.; and Altschul, A. M. Plant Physiol. 23: 84-97. 1948.

Previous experiments indicated the possibility of inhibiting by chemical treatment the biological activity that causes deterioration of seed during storage and demonstrated the need for new compounds that would be active in small concentrations. In the past only a few chemicals had been tried because of the length of time necessary to test their efficiency. In order to survey a wide variety of chemicals for inhibitory efficiency, a method was developed for rapidly evaluating a large number of potential inhibitors simultaneously. This method permits a quantitative evaluation not only of the biological activity but also of the quality of the seed after storage. By setting up the effect produced by one inhibitor as a standard and comparing the other chemicals to this standard, it was further possible to assign an inhibitor index to any chemical investigated. Application of this method in investigating the effects of ethylene chlorhydrin and vinyl propionate on heating and lipolysis in flaxseed is described in detail.

289. STORAGE OF COTTONSEED AND PEANUTS UNDER CONDITIONS WHICH MINIMIZE CHANGES IN CHEMICAL COMPOSITION. Stansbury, M. F.; and Guthrie, J. D. J. Agr. Res. 75: 49-61. 1947.

Analysis of cottonseed samples stored at room temperature, 0° C., and -18° C. showed that if dried to a moisture content of 8.3 percent or lower, cottonseed may be stored for more than a year in sealed containers at 1° C. or below without appreciable change in total nitrogen, total oil, free fatty acid content of oil, iodine number of oil, peroxide value of oil, or catalase activity of the kernel. Analysis of peanut samples stored at the three temperatures showed that unshelled peanuts may be stored for more than 2 years in closed cans at 1° or below without appreciable change in total nitrogen and oil contents of kernels, free fatty acid content, and iodine number of the oil.

259. BROMINE RETENTION BY COTTONSEED AND COTTONSEED PRODUCTS AFTER FUMIGATION OF THE SEED WITH METHYL BROMIDE. Reeves, W. A.; D'Aquin, E. L.; and Phillips, G. L. J. Am. Oil Chemists' Soc. 24: 217-19. 1947.

To determine the amount of bromine retained by seeds or by the oils obtained from the seeds after their fumigation with methyl bromide, three different fumigant treatments were applied to cottonseed having 5.2 percent moisture content. Seeds from each of the three treatments were processed in small-scale standard oil mill equipment using standard mill procedures. Samples of linters, hulls, meats, cake, and oil were analyzed for total bromine, and the values obtained are tabulated. The hydraulic-pressed oil from the fumigated cottonseed contained no bromine. The quantitative calculations of the recovery of bromine in the meats, also tabulated, show that the bromine in the meats appeared almost quantitatively in the press cake, further substantiating the conclusion that bromine compounds are not present in the oil obtained, and also indicating that the methyl bromide has reacted to produce nonvolatile bromine compounds. an indication supported by other data obtained.

252. INHIBITION OF HEATING AND LIPOLYSIS IN SEEDS. Condon, M. Z.; Andrews, F. R.; Lambou, M. G.; and Altschul, A. M. Science 105: 525-7. 1947.

Preliminary results are reported of a survey of the effectiveness of a wide variety of chemicals as inhibitors of the biological process responsible for the heating and lipolysis of

seeds during storage. The chemical is applied to artifically moistened seeds and its effect is determined by a calorimetric method. Response curves obtained in a typical experiment with moist flaxseed are given. A concentration of 0.24 percent diethyl oxalate inhibited heating as effectively as 0.38 percent ethylene chlorhydrin treatment. The comparative effectiveness of a series of chemicals is tabulated. The results show that a wide variety of chemicals in fairly low concentrations are capable of inhibiting heating and lipolysis in moist flaxseed; and a number of these chemicals have been tested and found equally effective in cottonseed and other seeds.

216. EFFECT OF INHIBITORS ON THE RESPIRATION AND STORAGE OF COTTONSEED. Altschul, A. M.; Karon, M. L.; Kyame, Lillian; and Hall, C. M. Plant Physiol. 21: 573-87. 1946.

To form a basis for a better understanding of the mechanism of inhibitor action, the effect of ammonia upon a number of properties of both mature and immature cottonseed was measured. To determine whether the surface sterilization of cottonseed would seriously inhibit respiration and lipolysis or affect the color of the oil, several other chemical agents known to inhibit microbial growth were also tested. Ammonia treatment of mature cottonseed inhibited the respiration and lipolysis and reduced the light absorption of the extracted oil at 360mμ. Similar treatment of immature cottonseed inhibited respiration but stimulated lipolysis and increased light absorption of the extracted oil at 560mμ. The vapors of Nacconol NR inhibited lipolysis under conditions where there was a stimulation of respiration. Treatment of cottonseed with 2'-methyl-1-maleanil yielded similar results. Emulsol 607M, Emulsion 607, and butyl-maleimide had no effect on lipolysis but stimulated respiration. Evidence is presented to demonstrate that most of the deterioration occurring in stored cottonseed is due to the action of enzymes in the seeds rather than to microbial activity.

204. AN APPARATUS AND METHOD FOR THE MEASUREMENT OF THE RESPIRATION OF COTTONSEED. Altschul, A. M.; Karon, M. L.; and Fynn, P. J. Plant Physiol. 21: 410-15. 1946.

A specially designed apparatus is illustrated whose use permits rapid and accurate measurement of the respiration rate of cottonseed. In the method of measurement, carbon dioxide and oxygen are successively removed from the gas sample, and the resulting changes in pressure of a fixed volume of the sample are used to calculate the percentages of carbon dioxide and oxygen in the sample. The procedure with a typical gas specimen is described, with tabulations of the results in the typical analysis. Results of all respiration experiments can be placed on a comparable basis by reducing the analytical values for carbon dioxide and oxygen to the number of cubic centimeters of gas, at standard temperature and pressure, evolved or consumed per day per gram of seed (moisture-free basis).

190. THE STORAGE OF COTTONSEED. IV. MILL-SCALE TESTS OF THE EFFECTS OF VARIOUS TREATMENTS OF COTTONSEED AND FLAXSEED ON HEATING AND FREE FATTY ACID FORMATION DURING STORAGE. Altschul, A. M.; Curet, M. E.; Karon, M. L.; Hall, C. M.; and Smith, B. A. Oil Mill Gaz. 50(8): 9, 11, 13, 23. 1946.

An ammonia treatment that had been successful in improving the storage properties of moist cottonseed in laboratory and small pilot-plant experiments proved to have no application, under present methods, when extended to mill-scale experiments. The vapors found in a commercial preparation of sodium alkyl aryl sulfonates in both laboratory and mill-scale tests inhibited heating and the rate of free fatty acid formation in cottonseed and flaxseed.

74. THE STORAGE OF COTTONSEED. III. FACTORS AFFECTING THE STORAGE OF MOIST COTTONSEED. Altschul, A. M. Oil Mill Gaz. 44(1): 8-9. 1944.

Biological processes of the cottonseed are outlined as an explanation of the effect of moisture in hastening deterioration. At a moisture content of 10.5 percent, 1.3 percent of free fatty acids was formed during storage for 160 days; by contrast, with a moisture content of 17 percent, 8.2 percent free fatty acids formed during storage for 129 days. Drying and cooling are suggested as methods for slowing down deterioration. A third method developed experimentally is described. This method consists of treatment with ammonia to inhibit respiration and the consequent heating and formation of free fatty acids. In storage tests, ammonia-treated seed with a moisture content of 15 percent showed formation of 4.8 percent free fatty acids, compared with 32 percent for untreated seed with a similar moisture content during a storage period of 180 days.

63. EFFECT OF MOISTURE AND OF TREATMENTS WITH ACID AND ALKALI ON RATE OF FORMATION OF FREE FATTY ACIDS IN STORED COTTONSEED. (Part I of a series on "The Storage of Cottonseed.") Karon, M. L.; and Altschul, A. M. Plant Physiol. 19: 310-25. 1944.

Cottonseed deteriorates during storage; free fatty acids increase, oil color darkens, protein is partly denatured, and viability decreases. The nature and mechanism of the various chemical systems responsible for these changes have been investigated. An equation was developed to express quantitatively the progress of hydrolysis of glycerides in cottonseed oil. The effect of the variation of moisture on the rate of formation of free fatty acids was determined over a moisture range of 7.5 to 20 percent (dry basis). Increase in moisture content results in an increased rate of hydrolysis of the glycerides. Treatment of the cottonseed with ammonia or hydrogen chloride gas retards the formation of free fatty acids. Three possible mechanisms of hydrolysis are suggested.

47. THE STORAGE OF COTTONSEED II. THE EFFECT OF AMMONIA TREATMENT ON THE FREE FATTY ACIDS AND COLOR OF THE SEED OIL, AND ON THE RATE AND DEGREE OF HEATING OF THE SEED. Altschul, A. M.; Karon, M. L.; Kyame, Lillian; and Caravelle, Maizie. Oil & Soap 20(12): 258-62. 1943. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-77, 8 pp. (Processed). 1945.

Treatment of moist cottonseed with ammonia prior to storage has been shown to reduce self-heating of the seed and the rate of formation of free fatty acids during storage. The color of oils obtained by solvent extraction from ammonia-treated cottonseed is considerably lighter than that of oils obtained from untreated cottonseed.

SEED STORAGE AND HANDLING - PATENTS

3326. COTTONSEED CLEANER AND FRACTIONATOR. Holzenthal, L. L.; and Gastrock, E. A. U. S. Pat. No. 3,261,466, July 19, 1966.

This invention provides an apparatus for cleaning and fractionating cottonseed and a cottonseed fractionating process for separating the particles normally present in cottonseed into a fraction consisting essentially of seed substantially free of foreign particles and, if desired, into several additional valuable fractions.

2954. APPARATUS FOR INDIVIDUALIZING COTTONSEED. Holzenthal, L. L.; Gastrock, E. A.; and Gentry, W. T. U. S. Pat. No. 3,169,277, February 16, 1965.

This invention relates to an apparatus for individualizing cottonseed and feeding the individualized cottonseed to a cleaning device that will subsequently separate the cottonseed from debris such as field trash, cottonseed stems, leaves, outer boll wall, shale, sand, and dirt. The apparatus that is the subject of this invention is designed as a piece of auxiliary equipment operating as an adjunct to a cottonseed cleaning device.

2057. COTTONSEED CLEANER. Holzenthal, L. L. U. S. Pat. No. 2,964,180, December 13, 1960.

A cottonseed cleaner useful for separating foreign matter from cottonseed. This cleaner consists of an endless, inclined moving belt with a resilient working surface. The resilient working surface of the belt is characterized by the presence of flexible protuberances. The protuberances are spaced closer together than the effective diameter of the seeds to be cleaned.

1326. STICK REMOVER. Holzenthal, L. L.; D'Aquin, E. L.; and Gastrock, E. A. U. S. Pat. No. 2,732,939, January 31, 1956.

A method for removing sticks and the like impurities from contaminated cottonseed, by (1) feeding the contaminated seeds into a substantially V-shaped crevice between a row of longitudinally advancing, substantially parallel strands, which are spaced apart just far enough for seeds to freely pass between them and the surface of a roll which is contacted by the rotates in conjuction with the advancing row of strands; and (2) separately collecting the partly purified seeds that pass through the row of strands and the stick-like impurities that move along with the advancing row of strands.

723. METHOD OF PREVENTING DETERIORATION OF SEEDS. Altschul, A. M.; Condon, M. Z.; and Lambou, M. G. U. S. Pat. No. 2,584,972, February 12, 1952.

The method is particularly applicable to seed, such as cottonseed containing natural enzymes and up to about 30 percent moisture, and comprises spraying the seed with a liquid chlorhydrin, such as ethylene chlorhydrin, in a concentration of from about 0.2 to 1.0 percent based on the dry weight of the seed.

680. METHOD OF PREVENTION OF DETERIORATION IN SEEDS. Altschul, A. M.; Condon, M. Z.; and Lambou, M. G. U. S. Pat. No. 2,571,095, October 16, 1951.

The biologically induced deterioration of cottonseed containing above 15 percent moisture is inhibited by contacting the seed with from 0.2 to 1.0 percent (by weight of dry seed) of a mixture of about 10 parts by weight propylene glycol dipropionate per part of 1,3-dimethyl-4,6-bischloromethylbenzene, and after about 1 to 3 days by reducing the moisture content of the seed below about 15 percent without the application of heat.

172. TREATMENT OF OILSEEDS. Altschul, A. M.; and Karon, M. L. U. S. Pat. No. 2,376,852, May 22, 1945.

The deterioration of cottonseed during storage is prevented by contacting the seeds with the vapors of a volatile base until the pH of the seeds is at least 8. The seeds so treated yield oils of an improved quality. This patent is particularly directed to the use of ammonia as the volatile base. U. S. Pat. 2,376,568 is directed to use of morpholine.

171. TREATMENT OF OILSEEDS. Altschul, A. M.; and Karon, M. L. U. S. Pat. No. 2,376,568, May 22, 1945.

Cottonseed deterioration during storage is prevented by contacting the seeds with the vapors of a volatile base until the pH of the seeds is at least 8. The seeds so treated yield an oil of an improved quality. This patent is particularly directed to the use of morpholine as the volatile base. U. S. Pat. 2,376,852 applies to a similar use of other volatile bases such as ammonia.

3091. APPLICATION OF CAPACITANCE METHOD FOR ANALYSIS, CONTROL, AND REMAKE OF SOLVENT MIXTURE IN THE MIXED SOLVENT EXTRACTION OF COTTONSEED. Cross, D. E.; Keating, E. J.; D'Aquin, E. L.; and Gastrock, E. A. Ind. Eng. Chem. Process Design Develop. 5: 94-97. 1966.

A method has been developed to analyze continuously the mixed solvent recovered from the hexane-acetone-water extraction of cottonseed and to correct automatically any deviation from the desired composition. This method, readily adaptable to existing plants, takes advantage of the fortuitous difference in dielectric constant between the components of the new solvent.

3049. A MIXED SOLVENT-EXTRACTION PROCESS FOR COTTONSEED. Gastrock, E. A.; D'Aquin, E. L.; Keating, E. J.; Krishnamoorthi, V.; and Vix, H. L. E. Cereal Sci. Today 10: 572-74, 576, 598-99. 1965.

The chemical engineering problems involved in developing the acetone-hexane-water (AHW) extraction process for cottonseed and progress toward their solution are presented. The AHW process involves many departures from orthodox solvent-extraction procedures based on commercial hexane. The problems involved are: (1) Removal with the oil of substantially all of the gossypol pigment present; (2) avoidance of excessive temperatures during processing to conserve the nutritive value of the extracted meal, especially its lysine content; (3) the presence of water in the solvent in significant percentages and its effect on solvent percolation, solvent drainage, and marc handling; (4) meal desolventizing; (5) oil desolventizing; (6) solvent recovery and remake; (7) mill-site refining of oils with high gossypol content; and (8) disposal of high gossypol content soapstock.

2684. AN ANALYSIS OF THE FILTRATION-EXTRACTION PROCESS FOR OILSEEDS. Gastrock, E. A. Oil Mill Gaz. 68(9): 14-17. 1964.

The major steps of the filtration-extraction process, the equipment used, and the sequence and cycle times of the various operations are reviewed. The advantages of the filtration-extraction process are summarized.

1686. MATERIAL BALANCES AT COTTONSEED OIL MILLS (Oil Mill Balances). Spadaro, J. J.; Hoffpauir, C. L.; Eaves, P. H., Jr.; Pominski, J.; and Gastrock, E. A. Oil Mill Gaz. 63(2): 37-39. 1958.

In an effort to find a solution to the "oil loss" problem a limited period mill-scale balance test was conducted at a commercial screw press-equipped mill. In this particular mill, under the processing conditions employed, about 2 pounds of oil per ton of cottonseed was not accounted for. This was not considered to be excessive.

1616. THE EFFECTS OF ALKALI COOKING ON THE YIELDS OF CRUDE AND NEUTRAL OIL FROM COTTONSEED MEATS. Eaves, P. H.; Molaison, L. J.; Knoepfler, N. B.; and Spadaro, J. J. J. Am. Oil Chemists' Soc. 35: 33-36. 1958.

Equal quantities of flaked cottonseed meats of identical composition were similarly cooked at high moisture conditions with and without alkali present. Analyses of the crude oils and meals were compared to determine the effects of the presence of alkali while cooking on the composition of the products. These experiments show a reduction in the yields of both crude and neutral oil resulting from the admixture of alkali with cottonseed flakes during cooking. Comparison is made between yields from flakes cooked with water and alkali-cooked yields. A similar comparison of the yields of neutral oil shows differences between those from alkali-cooked flakes and those cooked with water. Crude oils from alkali-cooked flakes contained

only about one-fifth as much gossypol as those from the water-cooked flakes and were appreciably lower in free fatty acids. Crude oil from the alkali-cooked flakes were significantly higher in phosphorus. The sodium content of the oils from alkali-cooked flakes indicated that their content of soaps ranged from 0.07 percent to 0.19 percent.

1464. FILTRATION-EXTRACTION PROCESS--COST ANALYSIS APPLICATION. Decossas, K.M.; Many, H.G.; McMillan, O.J., Jr.; Gastrock, E.A.; and Pollard, E.F. Ind. Eng. Chem. 49: 930-35. 1957.

The filtration-extraction process, a variation of the direct solvent process, was designed by the Southern Utilization Research and Development Division to provide the smaller cottonseed crusher with a more efficient and practical method of processing. This paper gives investment costs, operating expenses, and profit potential of hypothetical filtration-extraction mills, and costs for conversion from hydraulic and screw pressing to filtration-extraction in the North Delta (Arkansas) area for the 1954-55 season. Investment costs for the hypothetical filtration-extraction mills are in all cases less than those for comparable mills of the direct solvent or prepress solvent types; and increased profits indicate that conversion of hydraulic mills to filtration-extraction can be paid for in a few years.

1463. FILTRATION-EXTRACTION OF COTTONSEED OIL--A STAFF-INDUSTRY COLLABORATIVE REPORT. Haines, H.W., Jr.; Perry, G.C.; and Gastrock, E.A. Ind. Eng. Chem. 49: 920-29. 1957.

Today the larger hydraulic mills in this country are converting to solvent extraction at an increasing rate. Methods adopted include direct solvent, prepress solvent, and filtration-extraction. The latter method offers many advantages. A plant constructed for cottonseed processing will operate on soybeans by changing the feed stock. In the future, more adaptations of this process to other oleaginous materials, especially those of high oil content, can be expected. The filtration-extraction at the Mississippi Cottonseed Products Co. was the first large-scale installation on stream. The plant processes 140 to 200 tons of cottonseed per day or 80 to 130 tons of soybeans during alternate periods of operation.

1462. PILOT-PLANT DEVELOPMENT OF THE ALKALI COOKING PROCESS FOR COTTONSEED MEATS. II. EFFECT OF ADDITIONAL COMMINUTION. Knoepfler, N.B.; King, W.H.; Stansbury, M.F.; and McCourtney, E.J. J. Am. Oil Chemists' Soc. 34: 61-66. 1957.

Pilot-plant experiments were conducted to determine the effects of several methods of comminution of raw cottonseed meats, with and without alkali, upon the properties of the resulting meals. Comminution methods included grinding at moisture contents from 12 to 31 percent and rolling and rerolling at various moisture levels from 4 to 13 percent and at temperatures from 90 to 150° F. Moisture content appears to be critical; free gossypol content is inversely related to the moisture content during the rolling process. The nitrogen solubility is reduced slightly by rolling at moisture contents in excess of 10 percent. Mixing in the presence of alkali at moisture contents of 24 to 31 percent and at a temperature of 90° F. gave a significant reduction in nitrogen solubility; increased temperatures during flaking or mixing reduced free gossypol and also nitrogen solubility.

1461. PILOT-PLANT DEVELOPMENT OF THE ALKALI COOKING PROCESS FOR COTTONSEED MEATS. I. EFFECT OF FLAKE THICKNESS AND OF TIME, TEMPERATURE, AND MOISTURE CONTENT DURING COOKING. King, W.H.; Knoepfler, N.B.; Hoffpauir, C.L.; and Mc Courtney, E.J. J. Am. Oil Chemists' Soc. 34: 27-30. 1957.

Results of alkaline cooking experiments show that all of the four variables studied--cooking moisture content, temperature, time and flake thickness--influenced properties of the

resultant meals in varying degrees. Under the experimental conditions used, a high initial moisture content, about 18 percent, is necessary to lower the free gossypol content in the finished meal to less than 0.04 percent. When this amount of moisture is used, special equiment is required to break the pigment glands at a low temperature. In the presence of high moisture content and alkali the gossypol, which is released when the glands are ruptured, is bound to the meal. When sufficient initial moisture is present (24 to 31 percent), only traces of gossypol (0.10 percent or less) are found in the extracted crude oil. High temperatures in the presence of high moisture content reduce the solubility of the meal protein (in 0.02 N NaOH). Maximum nitrogen solubility obtained under conditions necessary to produce low free gossypol content was 64 percent. Finer comminution of the raw meats results in lower free gossypol content in the finished meal, in lower gossypol content of the oils and in more complete removal of the oil.

1460. COMPARATIVE ABSOLUTE YIELDS OF CRUDE AND NEUTRAL OILS FROM VARIOUSLY PREPARED COTTONSEED MEATS. Eaves, P.H.; Molaison, L.J.; Pollard, E.F.; and Spadaro, J.J. J. Am. Oil Chemists' Soc. 34: 156-59. 1957.

The method used in preparing cottonseed meats for extraction had a significant effect on the yields of crude oil obtained but the yields of neutral oil were virtually unaffected. The greatest yields of crude oil were obtained from raw flakes, intermediate yields from tempered flakes, and the smallest yields from cooked flakes. The impurities content in the respective crude oils followed the same order, i.e., crudes from raw flakes were highest in impurities and lowest in neutral oil, crudes from tempered flakes were lower in impurities and higher in neutral oil, and the crudes from the cooked meats were outstandingly low in impurities and high in neutral oil. Virtually equal amounts of neutral oil were obtained from equivalent quantities of comparable meats regardless of the method used in preparing the meats for extraction.

1384. FILTRATION-EXTRACTION PROCESS ACHIEVES COMMERCIAL STATUS. Gastrock, E.A. The Soybean Digest 16(12): 18-20. 1956.

Results of 2 years' experience in the operation of the filtration-extraction process on cottonseed and soybeans at the Mississippi Cottonseed Products Co. plant, Greenwood, Miss., are discussed. While some adjustments had to be made to adapt the pilot-plant operation to commercial scale, early claims for the process, based on the small-scale developments, were equalled or bettered. Filtration-extraction is a direct process. It does not depend on prepressing for efficient extraction of widely varying oilseeds, and offers a number of other advantages, particularly in the matter of economy of installation and operation. Data obtained from commercial operations on cottonseed and soybeans are included.

1307. SOLVENT COOKING OF COTTONSEED MEATS FOR EXTRACTION. Spadaro, J.J.; and Vix, H.L.E. J. Am. Oil Chemists' Soc. 33: 177-80. 1956.

Experiments indicate that solvent cooking offers the possibility of combining cooking, crisping, and extraction steps into a single operation in a process for the direct solvent extraction of cottonseed oil. Raw, rolled cottonseed meats are mixed in the solvent, together with sufficient moisture (containing added chemicals) and then the resulting slurry is subjected to a combined heating and azeotropic distillation, with further mixing, to reduce the moisture content to an optimum value. Addition of sodium hydroxide and calcium chloride improved agglomeration of meal particles and mass velocity, at the same time lowering the free gossypol content of the meal. Increase of moisture content resulted in mass velocities above the desirable 2,000 lbs. per square foot per hour, and the higher initial cooking moisture content, together with addition of chemicals, reduced the free gossypol content appreciably.

1303. EFFECT OF pH DURING THE COOKING OF COTTONSEED ON THE PROPERTIES OF MEALS AND OILS. King, W.H.; Wolford, L.T.; Thurber, F.H.; Altschul, A.M.;

Watts, A. B.; Pope, C. W.; and Conly, Jean. J. Am. Oil Chemists' Soc. 33: 71-74. 1956.

The cooking of cottonseed meats in the laboratory at varying pH levels, low temperatures, and high moistures resulted in improvement of the quality of both the meals and oils. The meals were high in soluble nitrogen, and at optimum pH levels their nutritive value, as measured by the growth rates of chickens in short-term tests, was superior to that of a butanone-extracted meal. Oils from meats cooked at low pH levels were high in gossypol and were subject to color reversion during storage, while those from meats cooked at high pH levels had a low refining loss, were low in gossypol, and were not subject to color reversion during storage. Both types of oils could be refined satisfactorily immediately after extraction. Parallelism between the curves showing the relationship between pH and nitrogen solubility and pH and nutritive value was marked.

1226. PREPARATION OF MEATS AND PROCESSING CONTROLS FOR LOW FREE GOSSYPOL AND HIGH PROTEIN SOLUBILITY. Gastrock, E. A.; Vix, H. L. E.; and D'Aquin, E. L. Oil Mill Gaz. 60(5): 12-13. 1955.

Free gossypol content and protein solubility in dilute alkali are two chemical indices used to measure or rate the toxicity and nutritive value of cottonseed meals. This paper is a review of various unit operations in cottonseed processing in order to show how they affect the free gossypol content and alkali solubility. Aside from general conditions, factors discussed are storage, conditioning and tempering, rolling or flaking, cooking, oil extraction, desolventizing, toasting, and degossypolization.

1223. MEASUREMENT OF EXTRACTABILITY OF RAW AND COOKED COTTONSEED FLAKES. Kulkarni, B. S.; D'Aquin, E. L.; and Graci, A. V., Jr. J. Am. Oil Chemists' Soc. 32: 691-97. 1955.

A batch cocurrent laboratory method for measuring comparative extraction rates and extraction efficiencies of oleaginous materials in solvent is described. The method, a modification of that by Wingard and Shard, was carefully tested with raw and cooked cottonseed flakes of various thicknesses up to .025 in., and in various hexane miscella concentrations up to 50 percent. It enables measurement of intrinsic extraction rates and extractabilities of materials, unaffected by diffusional effects in the liquid medium, and yields accurate and concordant results even with extracting miscellas of high concentration. The one-hour extraction test described is equally applicable for evaluating and predicting the effect upon extractability of different material preparation operations, particle sizes, moisture contents, temperature, and solvents.

1168. GENERAL DENSITY EQUATION FOR GLYCERIDIC OIL-SOLVENT MIXTURES. CALCULATION OF DENSITY-COMPOSITION-TEMPERATURE DATA FROM OIL AND SOLVENT DENSITIES. Skau, E. L.; Magne, F. C.; Mod, R. R.; and Durr, R. L. Ind. Eng. Chem. 47: 1043-49. 1955.

A simple quadratic equation has been derived that permits direct calculation of the density or the composition of any oil-solvent mixture, the temperature being known, from the densities of the oil and the solvent. The validity and general applicability of this equation have been established on the basis of extensive experimental data on 41 oil-solvent systems. The equation makes data for known and unknown systems readily available in any desired density or temperature units, eliminates graphical interpolations, and is applicable to all crude or refined animal, vegetable, and marine oils.

1158. PRESENT STATUS OF THE FILTRATION-EXTRACTION PROCESS FOR COTTONSEED. Spadaro, J. J.; Vix, H. L. E.; Gardner, H. K.; D'Aquin, E. L.; Eaves, P. H.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 32: 160-63. 1955.

Adaptation of the filtration-extraction process for cottonseed in the first commercial plant and improvements developed since publications of previous papers are presented. These include modifications in the method of conditioning of the meats or flakes; selection of suitable conveyors and filter medium; determination of optimum filter-cake thickness; and development of a method for clarification of the product miscella. Also discussed is the quality and color stability of the oil produced from comparable lots of cottonseed by the filtration-extraction process, as compared with that by hydraulic pressing, and by solvent extraction of uncooked flakes.

1157. PREPRESS-SOLVENT EXTRACTION OF COTTONSEED, PROCESSING CONDITIONS AND CHARACTERISTICS OF PRODUCTS. Pons, W. A., Jr.; Thurber, F. H.; and Hoffpauir, C. L. J. Am. Oil Chemists' Soc. 32: 98-103. 1955.

A study has been made of the relationship between processing conditions and the chemical characteristics of cottonseed meals and oils produced by prepress-solvent extraction. Cooking conditions are the major factor influencing the distribution of the gossypol between the meal and oil. Reduction in free gossypol during cooking is attributed to binding with meal components and that occurring during prepressing and solvent extraction mainly to removal of gossypol in the oils. Nitrogen solubility data indicate that the greatest protein damage occurred during cooking. Prepressed oils gave lower refining losses and lower refined and bleached color than did the solvent-extracted oils. Bleach-color reversion, after storage of crude oils for 30 days at 100° F., was greater for solvent-extracted than for prepressed oils.

1156. NOMOGRAPH FOR DETERMINING PERCENTAGE EXTRACTION EFFICIENCY. Graci, A. V., Jr. J. Am. Oil Chemists' Soc. 32: 109. 1955.

Convenient and accurate determinations of vegetable oil processing extraction efficiencies are made possible by a simple alignment chart. No mathematical calculations are required. The equation that forms the basis of the chart is given.

1153. A BENCH-SCALE METHOD FOR EVALUATING THE PROCESSING CHARACTERISTICS OF OILSEEDS FOR FILTRATION-EXTRACTION. Graci, A. V., Jr.; Spadaro, J. J.; Paredes, M. L.; D'Aquin, E. L.; and Vix, H. L. E. J. Am. Oil Chemists' Soc. 32: 129-31. 1955.

A simple laboratory testing apparatus has been devised to determine the extractability and filterability of oilseeds prepared for solvent extraction. By means of this apparatus and procedure it is possible, with a relatively small quantity of material, to evaluate many preparation and filtration variables and to predict with a fair degree of accuracy the performance of a continuous horizontal vacuum filter. It has been used for oilseeds, and could also be used for other materials where extraction and filtration operations are involved.

1099. SCREW PRESSING OF COTTONSEED. Wamble, A. C.; and Harris, W. B. Tex. Eng. Exp. Sta. Bull. No. 136. 1954.

Ways of improving press operations were sought. All work was carried out under controlled conditions, including full-scale operations in a manner applicable to commercial practice The main variables for each step have been analyzed for their effects upon extraction efficiency, quality of products, and other pertinent factors. Methods of improving oil yield, capacity, and all-around performance are described and the results projected as general principles for mill operation for comparison of screw press extraction with other methods.

1098. THE EFFECT OF PROCESSING CONDITIONS ON THE PROPERTIES OF SCREW-PRESS COTTONSEED MEAL AND OIL. Thurber, F. H.; Vix, H. L. E.; Pons, W. A. Jr.; Crovetto, A. J.; and Knoepfler, N. B. J. Am. Oil Chemists' Soc. 31: 384-88. 1954.

Four mill-scale studies were undertaken to produce quantities of low-temperature, dry-cooked meals and to obtain information that may aid in developing processing conditions that will retain certain desirable features of both low-temperature, dry cooking and high-temperature, moist cooking. Cooking meats at 240 to 250° F. produced meals with low free gossypol content, at the expense of considerable protein damage. The resultant crude oils showed some color reversion during storage at 95° F. Dry cooking (7 percent moisture) at temperatures not exceeding 200° F. gave meals of improved chemical properties, but the crude oils exhibited considerable color reversion in storage. Wet, low-temperature cooking (200 to 210° F.), followed by evaporative cooling, yielded a meal intermediate in quality between that for normal mill practice and dry, low-temperature cooking. The crude oils, which corresponded to hydraulic-pressed oil, did not exhibit any appreciable color reversion in storage at elevated temperatures. The increase in the bleach color of crude oils stored at 95 to 100° F. was directly related to the initial gossypol content of the crude oils. Nutritional studies on the meals were made by cooperating laboratories and are being reported separately.

1095. FILTRAXIONE-ESTRAXIONE: UN NUOVO PROCESSO DI ESTRAXIONE DIRETTA CON SOLVENTI DEI MATERIALI OLEOSI (FILTRATION-EXTRACTION: A NEW DIRECT SOLVENT EXTRACTION PROCESS FOR OLEAGINOUS MATERIALS.) Spadaro, J. J.; and Graci, A. V.; Jr. Olii Minerali, Grassi, Colori e Vernici. 6: 1-16. 1954.

This is an illustrated article giving details of procedures, equipment, results, and advantages of the filtration-extraction of cottonseed, rice bran, and soybeans.

1093. FILTRATION-EXTRACTION: A NEW COMMERCIAL CONTINUOUS SOLVENT-EXTRACTION PROCESS FOR EXTRACTION OF OLEAGINOUS MATERIALS. Kulkarni, B. S.; Graci, A. V., Jr.; and Vix, H. L. E. Oils and Oilseeds. J. 7(3): 5-11. 1954.

Briefly described is the case history of a novel solvent-extraction process from its conception to its operation of a commercial scale. Main advantages of the process over the conventional processes are summarized. Typical plant data have been included to confirm successful operation, and some of the novel design features of the process are illustrated.

1091. CURRENT STATUS OF THE FILTRATION-EXTRACTION PROCESS FOR COTTONSEED AND OTHER OLEAGINOUS MATERIALS. Gastrock, E. A.; Spadaro, J. J.; Gardner, H. K.; Knoepfler, N. B.; and Molaison, L. J. Oil Mill Gaz. 59(2): 40-41. 1954.

Early claims for this solvent process having been equalled or bettered by commercial operations at the Greenwood, Miss. plant processing soybeans and cottonseed. Further bench-scale work is being conducted on application of the process to rice bran, soybeans, cottonseed, flaxseed, and peanuts, and some has been done on milo germ and sesame; pilot-plant work is underway on rice bran, soybeans, cottonseed, flaxseed, and milo germ. A summary is given of the various processing operations in the filtration-extraction process and some general conclusions regarding the variable encountered and steps to be taken for controlling them.

1090. A NEW BATCH SOLVENT-EXTRACTION PILOT PLANT. Decossas, K. M.; Molaison, H. J.; and Pollard, E. F. J. Am. Oil Chemists' Soc. 31: 338-41. 1954.

A stainless steel, batch solvent-extraction pilot plant has been designed, constructed, and tested. This new improved batch solvent-extraction plant and its versatility of design are described. Data are given on the cost and initial test operations. The performance of the plant and its value as a research tool have been demonstrated by the production of free gossypol and meals low in gossypol for use in research. Only one operator is needed.

1032. LABORATORY EXPERIMENTS ON COOKING OF COTTONSEED TO PRODUCE IMPROVED MEAL AND OIL. Thurber, F. H. Oil Mill Gaz. 59(2): 72-74. 1954.

Experiments are underway to find a means of either removing gossypol from cottonseed meal or inactivating it during processing without lowering the nutritive value of the protein. In one study a meal of low gossypol content, prepared by extraction of oil with hexane followed by extraction of gossypol with methyl ethyl ketone, had exceptionally high protein efficiency in chick-feeding tests. This meal serves as a standard for determining the nutritive value of other cottonseed meals. Other studies deal with effects of moisture, temperature, and time on cottonseed flakes in a closed cooker without agitation, and effects of chemicals, particularly acids and alkalis, on the meats during cooking.

1015. WHEN SHOULD COTTONSEED OIL MILLS CONVERT? Gastrock, E. A. Cotton Trade J. Intern. Yearbook, 1953-54. Ed. 34(12): 36-38, 210. 1954.

Provided the proper local situation exists, conversion from hydraulic pressing to filtration-extraction can be considered, even for an 80-ton-per-day plant operating 150 days per year or more for larger installations. Conversion from screw pressing to filtration-extraction can be condisered for an 80-ton-per-day plant, especially if operating for 200 or 250 days per year. For the 150- and 200-ton-per-day plants the reasons for converting to filtration-extraction are stronger. Pay-out times for conversion from screw pressing are slightly longer than for conversion from hydraulic pressing, the differences varying from 0.9 to 2.7 years. This article gives four tables of data to show comparative operating costs for hydraulic pressing, screw pressing, and solvent extraction; oil gains per ton of cottonseed crushed by conversion to filtration-extraction; annual gains and pay-out times for plants operating over seasons of different lengths.

1014. SEAL A VACUUM SOLVENT SYSTEM WITH THE SOLVENT. Gastrock, E. A. Ind. Eng. Chem. 46(3): 124A. 1954.

In connection with pilot-plant solvent-extraction of oilseeds, a novel solution to the problem of using vacuum in systems involving volatile and flammable solvents was found. Solvent is used as the sealing liquid with centrifugal vacuum pumps. The scheme is being used commercially in a plant for filtration-extraction of cottonseed recently put into operation by Mississippi Cottonseed Products Co., Greenwood.

950. PHASE EQUILIBRIUM DATA PERTAINING TO THE EXTRACTION OF COTTONSEED OIL WITH ETHANOL AND 2-PROPANOL. Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 30: 288-91. 1953.

Basic solubility data have been obtained pertaining to the use of ethyl and isopropyl alcohols in the extraction of oil from cottonseed. This information augments data reported by others covering only a part of the range, and is based in part on titration methods. These combined data will assist in selection of the most desirable temperature and moisture concentrations in the solvent extraction of cottonseed with these alcohols. Comparison with results previously published for soybean oil suggests that the mutual solubility data for cottonseed oil and aqueous ethanols are applicable to other vegetable oils over a wide range of iodine values.

943. USE OF PREPARATION TECHNIQUES TO OVERCOME COTTONSEED VARIABLES. Gastrock, E. A. Oil Mill Gaz. 58(1): 34-37. 1953. (Brief version, Gastrock, E. A.; and Persell, R. M.) Publ. Oil Mill Gaz. 57(12): 27.1953.

Some variables encountered in the raw cottonseed are described. It is pointed out that preparation operations are a means of solving some of the problems involved. The continuing object of researchers and mill superintendents is to make it possible to process material such as cottonseed through any one of the five different commercial processes for recovery of oil to produce substantially uniform products of the best quality and to provide lowest cost compatible with each installation.

941. PROCESSING OF COTTONSEED. I. REVIEW OF RESEARCH TO IMPROVE NUTRITIVE VALUE OF MEAL AND COLOR OF OIL. II. PRELIMINARY REPORT OF RESULTS OF LABORATORY RESEARCH. Altschul, A. M.; and Thurber, F. H. Cotton Gin & Oil Mill Press 54(23): 26, 68-71. 1953.

Problems involved in improving the quality of cottonseed meal and oil and reports of research to aid in solving these problems are reviewed. Many papers that resulted from this program of research are listed in a bibliography. Some of the laboratory work suggests possibilities for modifying processing conditions to improve the quality of meal and oil.

918. AN INVESTIGATION OF SOME FACTORS AFFECTING THE EFFICIENCY OF HYDRAULIC PRESSING OF COTTONSEED. Carter, C. L. Tenn. Univ. Eng. Expt. Sta. Bull. No. 18. 1953.

This report describes, in part, an investigation carried out between 1949 and 1952, setting forth conclusions, recommendations for further work, and recommended pressroom operating methods for hydraulic press mills. A thorough and systematic study was made of pressroom variables, using bench-scale laboratory equipment. The validity of the results was tested by performing a limited number of experiments in an operating mill. The primary object was to determine the effect of pressing variables (total pressure applied, rate of application of pressure, pressing time, pressing temperature, moisture content and hull content, and type or variety of seed) on the amount of oil removed from the cooked meats. A secondary object was to determine the effect of these variables on the quality of the oil and meal.

879. GOSSYPOL MATERIAL BALANCE, DENATURATION OF PROTEIN, AND LOSS OF THIAMINE IN COMMERCIAL PROCESSING OF COTTONSEED. Pons, W. A., Jr.; Murray, M. D.; Leblanc, M. F. H., Jr.; and Castillon, L. E. J. Am. Oil Chemists' Soc. 30: 128-32. 1953.

Cottonseed processed by five commercial mills was systemically examined with reference to free gossypol reduction, nitrogen solubility, thiamine reduction, total gossypol, and the distribution of gossypol in processing. One hydraulic mill reduced free gossypol in meal to a low level, about that obtained in screw pressing. For a given mill free gossypol contents of meals were fairly uniform. A relatively small amount of total gossypol was lost or destroyed during preparation of meats for either hydraulic or screw pressing; no significant loss could be attributed to pressing operations. Screw-pressed oils appear to contain several times as much gossypol as hydraulic pressed oils, the amount depending on the extent of binding of gossypol in cooking and mechanical preparation of meats. The high temperatures developed in screw pressing contributed to a greater reduction in thiamine and nitrogen solubility than was observed for hydraulic pressing.

877. RESEARCH ON THE PREPARATION OF COTTONSEED MEATS FOR FILTRATION-EXTRACTION. Knoepfler, N. B.; Eaves, P. H.; and Vix, H. L. E. Cotton Gin & Oil Mill Press 54(6): 98-100. 1953.

Cottonseed meals produced experimentally by different methods of processing are being used in various nutritional investigations. Conditions in the rolling and cooking operations to prepare cottonseed meats for filtration-extraction have been studied in relation to the quality of the final meal and to the yield and quality of the oil. The preparation of cottonseed meats for filtration-extraction was compared with that for hydraulic pressing.

873. DIVIDING OIL MILL OPERATIONS TO BOOST PROFITS SAID HELPFUL. Gastrock, E. A.; and Persell, R. M. Cotton Trade J. 33(19): 8 1953. (Also in Oil Mill Gaz. 57(12): 27. 1953; erroneously under the title of another article, Use of Preparation Techniques to Overcome Cottonseed Variables.)

The process engineer seeking to increase oil mill profits will find it convenient to divide the operations of such a mill into purchasing the seed, processing the seed into oil, meal, hulls, and linters of a marketable quality, and selling these products at a profit. Factors that determine profits are: The relation between seed costs and product prices; volume of the seed processed; and performance of the mill. An analysis of these factors has been found useful in cottonseed processing at the Southern Regional Research Laboratory.

854. SOLVENT EXTRACTION OF OIL FROM COTTONSEED PRIOR TO THE REMOVAL OF LINTERS AND TREATMENT OF THE RESIDUE TO EFFECT SEPARATION OF MEAL, HULLS AND LINTERS. Clark, S. P.; and Wamble, A. C. Texas Eng. Expt. Sta. Bull. 125. 1951. (Summary, including later information, J. Am. Oil Chemists' Soc. 29: 624-26. 1952.)

Mechanical removal of linters from cottonseed before solvent extraction of oil is one of the most expensive operations in cottonseed processing. A commercially feasible method was sought that would avoid or reduce expense, yield cleaner linters, and save some of the oil commonly lost with hull fragments. Details of the methods tried, and the results, are reported.

853. A LABORATORY APPARATUS FOR DETERMINING THE RATE OF EXTRACTION OF OIL FROM OIL-BEARING MATERIALS. Clark, S. P.; and Wamble, A. C. J. Am. Oil Chemists' Soc. 29: 56-59. 1952.

Equipment and a method have been described for measuring the rate of extraction of oil from oil-bearing materials by solvent. The method consists essentially of immersing wire baskets containing the oil-bearing material in a circulating stream of solvent for varying periods of time. After oil is extracted, the baskets are centrifuged to remove excess solvent and the residual oil is determined. The method is patterned after one devised by Boucher et al. but has been modified and extended to other oil-bearing materials.

852. A PRELIMINARY STUDY OF SOME FACTORS AFFECTING THE HYDRAULIC PRESSING OF COTTONSEED. Carter, C. L. Oil Mill Gaz. 56(8): 84-86, 88-89. 1952.

A number of factors affecting the efficiency of hydraulic pressing of cottonseed have been studied by using a small laboratory cooler and a battery of Carver laboratory presses. Meats were cooked at 220° F. and at atmospheric pressure for one hour. Cubes of about 3/4-inch size were formed at different pressing moisture contents. Pressing temperatures of 140 to 235° F. were maintained by circulating oil in jackets around the press rams and in hollow platens on the rams. Under one set of pressing conditions an increase in pressing temperature from 130 to 230° F. resulted in a decrease in minimum residual oil from 5 percent to less than 4 percent (dry, oil-free basis). It was found that the shape and position of the curve of residual oil vs. moisture are altered appreciably by changes in pressing temperature, rate of application of the pressure, and hull content of the meats. Effects on residual oil vs. moisture relationship of pressing temperature, total pressure, rate of application of pressure, and hull content of the meats are not mutually independent.

842. ELECTRICAL SYSTEM IN SOLVENT AREA. Decossas, K. M.; Molaison, H. J.; Deckbar, F. A., Jr.; and Asset, H. L. South. Power & Ind. 70(11): 76, 80. 1952.

The electrical system of an indoor pilot-plant solvent area at the Southern Regional Research Laboratory, covering 5,000 sq. ft. served entirely with Class 1, Group D Fixtures, is described. The area comprises four oilseed processing plants: A continuous solvent-extraction plant; a continuous solvent crystallization plant; an oil-and-solvent-recovery plant; and a cottonseed fractionation plant. The overall cost of the electrical system is given.

810 A PRELIMINARY COST STUDY OF THE FILTRATION EXTRACTION OF COTTONSEED. Persell, R. M.; Pollard, E. F.; Deckbar, F. A., Jr.; and Gastrock, E. A. Cotton Gin and Oil Mill Press 53(17): 18, 20. 1952.

This study indicates that the cost of a filtration-extraction plant processing 100 tons of cottonseed a day would be in line with that of a comparable conventional direct solvent-extraction plant, with some savings in favor of the filtration-extraction plant. A high degree of mechanization and low supervision and operating costs can be anticipated in filtration-extraction plants. The production of high quality oil and meal will insure maximum returns.

809. BREAK-EVEN CHART VALUABLE IN OPERATION OF OIL MILL. Gastrock, E. A.; Persell, R. M.; and Pollard, E. F. The Cotton Trade J. 34(49): 6. 1952.

In a simple, graphical method of analyzing the operating results of an oil mill, a chart shows the point where the total costs line crosses the total income line. This is the break-even point.

808. MATERIAL BALANCES FOR FILTRATION-EXTRACTION AND FOUR CONVENTIONAL METHODS OF PROCESSING COTTONSEED. Gastrock, E. A.; Eaves, P. H.; and D'Aquin, E. L. Oil Mill Gaz. 57(1): 62-63. 1952.

Comparative material balances for filtration-extraction and for hydraulic pressing, screw pressing, direct solvent extraction, and prepress solvent extraction are presented to illustrate differences of the processes in yields of products, processing costs, and fixed costs.

807. OPERATING FEATURES OF FILTRATION-EXTRACTION AND OTHER SOLVENT-EXTRACTION PROCESSES. Gastrock, E. A.; D'Aquin, E. L.; and Spadaro, J. J. Oil Mill Gaz. 57(1): 26-29. 1952.

The unit operations required for each process, material residence time for each, the sequence of these operations, and the cumulative material residence time for both the preparation and the extraction phases are compared for filtration-extraction and for conventional direct and prepress solvent processes. Filtration-extraction, by utilizing cooking, does not require meticulous preparation of flakes. While prepress extraction recovers slightly more oil, filtration-extraction eliminates the need for pressing, oil screening, grinding, conditioning, and the power and equipment required for conducting these operations.

806. FLAKE FEEDING DEVICE FOR SOLVENT-EXTRACTION OF OIL-BEARING MATERIALS. Gardner, H. K.; D'Aquin, E. L.; Parker, J. S.; and Gastrock, E. A. Ind. and Eng. Chem. 44: 2261-64. 1952.

A device was developed to feed flaked oil-bearing materials to a pilot-plant size solvent extractor; to provide a continuous, uniform discharge of the material, which can be varied over a 2.5 to 1 range; to form a positive seal plug to prevent the escape of solvent vapors at the point of entry of the material; and to cause a minimum breakage of the material into very small particles. The device operated satisfactorily with flakes from cottonseed, peanuts, okra seed, and rice bran, and it is suited for feeding a wide variety of materials other than oilseed flakes or meats. Scaling up to commercial size should be feasible.

805. A REVIEW OF THE COMMERCIAL AND EXPERIMENTAL PROCESSING OF OIL-BEARING MATERIALS. Deckbar, F. A., Jr.; Persell, R. M.; Pollard, E. F.; and Gastrock, E. A. Cotton Gin & Oil Mill Press 53(15): 13-15, 17, 48, 50-51, 54-56, 58-62. 1952.

Information is given on the processing of 43 oil-bearing materials, with statistics on their production, oil content, and uses. The materials are discussed in the categories of: available

domestically and commercially processed; important in world trade and imported only; processed abroad, available domestically, but processed either not at all or little; under domestic agricultural development, but as yet processed either in the pilot plant or on a limited commercial scale. In the last category, sesame, safflower, and sunflower seed and castor beans seem to be increasing in importance.

730. RESEARCH ON PROCESSING PROCEDURES TO IMPROVE THE NUTRITIVE VALUE OF COTTONSEED MEAL. Knoepfler, N. B.; Vix, H. L. E.; and Thurber, F. H. Cotton Gin & Oil Mill Press 53(6): 61-66. 1952.

Progress made in an investigation of the processing conditions suitable for the production of cottonseed meals of high nutritive value by fractionation, screw pressing, hydraulic pressing, and solvent extraction is reported. This work was part of a broad program of revaluation of the nutritional value of cottonseed meals as affected by processing procedures, which is being conducted by this Laboratory in cooperation with the National Cottonseed Products Association, oil mills, and State, Federal, and private laboratories.

728. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. IX. DETERMINATION OF FINES IN MISCELLA. Graci, A. V., Jr.; Crovetto, A. J.; Parker, J. S.; and Reuther, C. G., Jr. J. Am. Oil Chemists' Soc. 29: 71-73. 1952.

Cottonseed, peanuts, okra seed, and rice bran were used in experiments to develop a rapid method of determining the solids content of oil-solvent miscellas. The method is volumetric and replaces the slower gravimetric method. The volumetric determination is converted to weight values by the use of an appropriate curve. The construction of the curve and its application in pilot-plant operations are described.

727. PILOT PLANT DESOLVENTIZATION OF FINE COTTONSEED MEAL. Graci, A. V., Jr.; Gardner, H. K., Jr.; Cucullu, A. F.; Crovetto, A. J.; Spadaro, J. J.; and Knoepfler, N. B. J. Am. Oil Chemists' Soc. 29: 41-43. 1952.

Fine cottonseed meal, 300-mesh, was desolventized satisfactorily in a continuous pilot-plant meal dryer of the screw-conveyor type. The free gossypol content of the fine meal was lowered as much as 69 percent during the drying operation without appreciably affecting the protein solubility of meal. A meal in granular form could be obtained, provided the feed material had certain physical characteristics.

726. FILTRATION EXTRACTION: A SIMPLIFIED SOLVENT EXTRACTION PROCESS. Gastrock, E. A.; D'Aquin, E. L.; and Vix, H. L. E. (Proc. 56th Ann. Conv. Natl. Cottonseed Prod. Assoc. 1952; 30, 32, 34, 36, 37. Publ. Cotton Gin & Oil Mill Press. Unnumbered.)

Described is filtration extraction, a new process for extracting cottonseed, which is based on the use of a continuous, horizontal, vacuum filter, in which the oil is separated after cooked flakes have been soaked in a separate vessel in contact with the solvent. Flakes are conditioned with shorter cooking periods and at somewhat lower temperatures than usually used, the maximum being about 230° F. Filtration extraction apparently overcomes most of the problems inherent in conventional direct solvent extraction and in solvent extraction with prepressing. The process is promising for application to soybeans, peanuts, rice bran, and other oilseeds.

724. A COMPARISON OF FIVE COMMERCIAL SOLVENTS FOR EXTRACTION OF COTTONSEED. Eaves, P. H.; Molaison, L. J.; Black, C. L.; Crovetto, A. J.; and D'Aquin, E. L. J. Am. Oil Chemists Soc. 29: 88-94. 1952.

In pilot-scale batch extraction of cottonseed, a comparison was made of benzene, ethyl ether, acetone, and butanone of commercial grade with commercial hexane as to effects on yield, composition, properties, and processing characteristics of the meals and the crude oils produced. None of the experimental solvents may be said to compare favorably with hexane, since any improvement in meal quality was achieved at the expense of oil quality, and the differences in yields were not sufficient to offset this loss in oil quality.

694. COTTONSEED PROCESSING RESEARCH AT THE TEXAS ENGINEERING EXPERIMENT STATION. Melloh, A.W. Oil Mill Gaz. 56(2): 11-15, 17-20. 1951.

The program of research in cottonseed processing and utilization at the Texas Engineering Experiment Station is reviewed. One section is devoted to a summary of the work done on "Solvent Extraction of Oil from Cottonseed Prior to the Removal of Linters, and Treatment of the Residue to Effect Separation of Meal, Hulls and Linters." This research was carried out under contract with the Southern Regional Research Laboratory.

663. PRE-PILOT PLANT INVESTIGATION OF A SOLVENT EXTRACTION METHOD FOR COTTONSEED BASED ON REDUCED PRESSURE FILTRATION. Spadaro, J.J.; Graci, A.V.; Gardner, H.K.; Parker, J.S.; Laborde, E.J.; and Gastrock, E.A. Oil Mill Gaz. 56(1): 77-81. 1951.

A pre-pilot plant investigation of a new method of solvent extraction presenting numerous potential advantages is reported. Several methods of preparation of cooked and uncooked flakes are described. Filtration rates, miscella concentrations, extractabilities, and capacities per square foot of filtering area are given, and effects of cake thicknesses and slurrying time are illustrated by graphs. A miscella with a high concentration of oil and an extracted cake with low residual oil and low solvent content were obtained. A flow diagram of the process is included.

652. RECENT TECHNOLOGICAL ADVANCES IN COTTONSEED PROCESSING. Gastrock, E.A. Oil Mill Gaz. 56(1): 35-37. 1951.

Recent technological advances in cottonseed processing by hydraulic pressing, screw pressing, and solvent extraction are briefly reviewed. Problems that still require solution include the need to increase oil yields with hydraulic pressing, to improve the quality of the oil by screw pressing, and to overcome the difficulties in preparing material for solvent extraction. A simplified solvent extraction process, using filtration as the major unit operation, and research in which much has been accomplished to improve product quality are described in detail.

645. THE EFFECT OF SCREW-PRESS AND HYDRAULIC-PRESS PROCESSING CONDITIONS ON PIGMENT GLANDS IN COTTONSEED. Batson, D.M.; Thurber, F.H.; and Altschul, A.M. J. Am. Oil Chemists' Soc. 28: 468-72. 1951.

A procedure is given for estimating the amount of intact pigment glands in cottonseed kernels and meal. Amounts of intact pigment glands, of free and total gossypol in uncooked and cooked meats, and press cake samples from laboratory-scale tests, from hydraulic-press mills, and from a screw-press mill were determined. An important finding was that in the screw-press mill, where cooking was carried out without the addition of moisture, little change in percentage of recoverable glands or of free gossypol occurred during cooking, but both components were reduced to very low levels during passage of the cooked meats through the screw press. In laboratory tests, when meats that had been cooked at low moisture content were subjected to hydraulic pressures of 2,000 and 20,000 p.s.i., the percentage of recoverable glands was reduced, but no corresponding reduction in free gossypol was found. Wet cooking of meats reduced percentages of free gossypol and intact glands and, although hydraulic pressing failed to further reduce the free gossypol, the percentage of recoverable glands was sharply reduced by pressing at both levels.

588. SEPARATING THE FRACTIONS OF COTTONSEED. Pollard, E.F.; Vix, H.L.E.; and Spadaro, J.J. Yearbook Agr., U.S. Dept. Agr. 1950-51. 561-65.

Two methods developed for the separation, or fractionation, of cottonseed-flake components are described. One, the flotation process that takes advantage of the differences in densities

of the pigment glands, meal, and hulls (the solid parts of cottonseed) was shown in extensive tests to have inherent disadvantages for commercial adoption. As a result, the second fractionation process, called the differential-settling process, was developed. This process depends principally on the force of frictional resistance between the solvent and the solid components in the slurry. The steps followed in the pilot-plant procedure are enumerated and discussed, and the process is evaluated from the practical standpoint.

582. RECOVERING OIL AND MEAL. D'Aquin, E. L.; Gastrock, E. A.; and Brekke, O. L. Yearbook Agr., U.S. Dept. Agr. 1950-51. 504-12.

The three methods in common use to separate oil from meal in oilseeds--hydraulic pressing, screw pressing, and solvent extraction--are differentiated, and the extent to which each is now used is discussed. The general steps required to prepare oilseeds for the removal of oil are discussed. Details vary with the particular oilseeds and the particular processes. Some technological considerations in each of three methods of processing oilseeds are discussed. Details are given for the solvent-extraction processes. Some advantages and disadvantages of the three processes for operators of oil mills are mentioned.

562. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. VIII. EFFECTS OF MOISTURE ON THE PREPARATION AND FLAKING OF COTTONSEED. Reuther, C. G., Jr.; Westbrook, R. D.; Hoffman, Wade H., Jr.; Vix, H. L. E.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 28: 146-49. 1951.

The feed material is of primary importance in the preparation of cottonseed for solvent extraction. Effect of moisture content of the delintered seed on the preparation of flakes having relatively few fines and allowing the ready percolation of solvent is investigated and evaluated. The work consisted of hulling, separating, and flaking in the pilot plant delintered seed of different moisture levels and then evaluating in the laboratory the flakes produced. The results indicate that 9 to 10 percent moisture in the meats is the best level for the preparation of flakes of the desired characteristics.

557. RESEARCH ON FRACTIONATION OF COTTONSEED MEATS. Knoepfler, N. B.; Graci, A. V., Jr.; Spadaro, J. J.; and Gastrock, E. A. Oil Mill Gaz. 55(8): 66-71. 1951.

Progress in developing a process for fractionation of cottonseed has been made during the past year and is reported. Advances were made in developing continuous pilot-plant unit operations to replace batch operations. Preparation factors affecting fractionation methods of obtaining greater reduction of gossypol were further investigated. Products from fractionation are being evaluated.

504. PILOT-PLANT FRACTIONATION OF COTTONSEED. III. PROCESS DEVELOPMENT OF DIFFERENTIAL SETTLING. Spadaro, J. J.; Persell, R. M.; Reuther, C. G., Jr.; Vix, H. L. E.; Laborde, E. J.; Latham, J. W.; Jaeger, R. L.; Pollard, E. F.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 27: 336-43. 1950.

Chemical engineering data are presented to show the pilot-plant development of cottonseed fractionation employing the differential settling principle. The purpose of the process is to produce one cottonseed meal fraction essentially free of pigment glands and hulls and a second fraction in which the pigment glands are concentrated sufficiently to serve as a raw material if pharmaceutical or other industrial use is developed for the glands or the pigments. The nonlipids fraction will make available a meal of high nutritive value and a source of industrial protein. Unit operations involved, including machinery and other equipment required, and proposed flow diagram for commercial application are discussed.

503. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. VII. EFFECT OF DRYING FLAKED PRIME COTTONSEED ON COLOR OF OIL AND MEAL PROPERTIES. Spadaro, J. J.; McCourtney, E. J.; and Vix, H. L. E. J. Am. Oil Chemists' Soc. 27: 394-96. 1950.

Effects of drying cottonseed flakes for solvent extraction on properties of oil and meal have been investigated. The seed had 6.9 percent initial moisture content and 1.2 percent free fatty acid content. Although the color increased slightly with increased drying temperatures of the flakes, all of the oil colors were prime. This indicates that dark oils produced by solvent extraction from this lot of cottonseed are due to conditions other than flake drying temperatures. The investigation indicated that prime flakes can be dried at relatively high temperatures without affecting the protein solubility of the meal. Proper drying of flakes will aid in obtaining optimum solvent extraction conditions, such as percolation rate, extractability of flakes, and minimum fines.

500. PILOT-PLANT FRACTIONATION OF COTTONSEED. V. A PRELIMINARY COST STUDY. Persell, R. M.; Vix, H. L. E.; Reuther, C. G.; and Pollard, E. F. J. Am. Oil Chemists' Soc. 27: 383-86. 1950.

A preliminary cost study of a proposed combination screw-press-fractionation plant showed that the economic advantage of the combination plant over the screw-press operation is small in comparison with the large additional investment that would be required for the combination plant. The present value of the combination process lies in the production of two new products--a purified high-protein meal and a concentrated pigment gland fraction--and in the possibility of producing a high-grade oil by the removal of the whole pigment gland, as is done in the fractionation process. Pilot-plant work underway indicates improvements in the fractionation process such as a higher recovery of still more improved meal.

459. RESEARCH ON FRACTIONATION OF COTTONSEED MEATS. Reuther, C. G., Jr.; Spadaro, J. J.; and Gastrock, E. A. Cotton Gin & Oil Mill Press 52(2): 12, 35, 39-40. 1950.

Progress in the development of a process of fractionation of cottonseed meats is reviewed, with emphasis upon the contribution of the National Cottonseed Products Association Fellow to these investigations. The fractionation process has been improved to give increased yields of fine meal, essentially free of pigment glands. Feed flakes suitable for partial solvent extraction prior to fractionation have been produced and sufficient quantities of products have been made for evaluation on a larger scale. In the fractionation process, either the defatted or undefatted flakes are disintegrated in a solvent slurry to detach the meal tissue from the pigment glands. The disintegrated solids are then separated by either the gravity flotation method or the differential settling method; and the solids fractions are recovered from the oil-solvent mixtures (miscellas), which are, in turn, evaporated to recover the oil and solvents. Before the process can be considered for commercial adoption, flake preparation, disintegration and screening procedures, and product evaluation must be further investigated.

457. INSTRUMENTATION FOR PILOT-PLANTS: OPERATION AND APPLICATION FUNDAMENTALS. Pollard, E. F.; Persell, R. M.; Molaison, H. J.; and Gastrock, E. A. Ind. Eng. Chem. 42: 748-52. 1950.

Instrumentation fundamentals are presented as a guide to chemical engineers in the application of instrumentation in pilot plants. The elements of control systems and the control characteristics of processes are defined. The operation and application of simple control systems are illustrated and discussed. Several automatic controller actions in industrial use are described in detail. The terminology, definitions, and illustrations used are familiar to chemical engineers and will give them an initial orientation in this field.

455. PILOT-PLANT EQUIPMENT COSTS. Molaison, H. J.; Deckbar, F. A.; and Cook, N. C. Chem. Eng. 57(4): 110-11. 1950.

A survey of pilot-plant process equipment purchased over a 6-year period at the Southern Regional Research Laboratory is briefly reported. The survey covers only equipment of carbon steel construction that was designed by engineers of the laboratory and for which detailed drawings were furnished to prospective bidders. The equipment is grouped into four general classifications and its cost calculated in cost per pound. The averages and ranges of unit cost for the four general types of equipment are tabulated. A procedure for using the data is recommended.

417. PILOT-PLANT FRACTIONATION OF COTTONSEED. IV. A REVIEW OF PROGRESS. Gastrock, E. A.; Persell, R. M.; Spadaro, J. J.; and Vix, H. L. E. Oil Mill Gaz. 54(3): 11-19. 1949.

Since the Southern Regional Research Laboratory was established, its workers have actively investigated the composition, properties, and characteristics of cottonseed pigments in an effort to find some solution to the processing problems occasioned by them. This article reviews the progress of research from the isolation of pigments to the development and improvement of a pilot-plant process for fractionating cottonseed into oil, meal, and pigment glands. Recovery of up to 70 percent of the available meal in cottonseed meats as light-colored, fine meal, essentially free of pigment glands, is now possible. The coarse meal fraction, containing about 10 percent pigment glands, would be the logical starting raw material for any industrial utilization of pigment glands or gossypol, but requires further treatment for use in regular commercial outlets. Present studies are directed towards reducing the percentage of coarse meal and increasing the percentage of purified fine meal. Cost estimates are being made for comparison with those of other processing methods.

400. PROCESSING OF COTTONSEED. IV. EFFECT OF PREPARATION AND COOKING OF MEATS ON THE BLEACH COLOR AND STORAGE PROPERTIES OF SCREW-PRESSED OILS. Williams, P. A.; Hadden, R. P.; Hall, C. M.; Castillon, L. E.; Guice, W. A.; O'Connor, R. T.; and Boatner, C. H. J. Am. Oil Chemists' Soc. 26: 28-34. 1949.

In order to study their effect on the characteristics of cottonseed meal and oil produced by screw pressing, conditions of processing were varied with respect to preparation of meats, addition of water, and the temperature and duration of cooking. In laboratory experiments, 10 percent of water added to the flaked meats before cooking at 235 to 244° F. for 1-1/2 hours resulted in a low bleach color in the expressed oils. Mill-scale tests indicated that cooking in the presence of relatively large amounts of water improves the oil's storage properties but not its initial bleach color. On the other hand, unwetted rolled or ground meats cooked at a low temperature resulted in low initial bleach color of the oil with very little reversion during storage at room temperature. Widely varying amounts of gossypol were found in both oils and meals produced from meats cooked under various conditions.

356. PILOT-PLANT FRACTIONATION OF COTTONSEED. I. DISINTEGRATION OF COTTONSEED MEATS. Spadaro, J. J.; Persell, R. M.; Murphey, C. H., Jr.; Vix, H. L. E.; McCourtney, E. J.; Hecker, J. L.; Pollard, E. F.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 25: 345-53. 1948.

A thorough investigation of the engineering problems encountered in all stages of the recently described fractionation method of processing cottonseed has been started. One of the first steps in this process is the disintegration of cottonseed meats to detach meal tissue from the pigment gland, making separation of the whole gland possible. The authors present results of tests utilizing a high-speed, "dissolver-type" impeller for disintegration by liquid shear. More than 90 percent of through-80-mesh material was obtained by this type unit. Moisture content

above 5 percent appreciably reduced the efficiency of disintegration and also increased power consumption and the viscosity of the slurry. Whole flakes resulted in a finer product than flakes prepulverized in the dry state. It was found that the presence of hulls slightly increased disintegration, power consumption, and viscosity; that higher speeds (up to 6,000 f.p.m. peripheral speed) were more efficient; and that the effect of solvents was small.

353. CONTINUOUS SOLVENT EXTRACTION OF COTTONSEED AND PEANUTS AT THE SOUTHERN REGIONAL RESEARCH LABORATORY. Gastrock, E.A.; and D'Aquin, E.L. Oil Mill Gaz. 55(4): 13-21. 1948.

The Southern Regional Research Laboratory's pilot-plant equipment for continuous solvent extraction of oilseeds is described, and the course of the solvent and meal in the process is diagrammed. Various difficulties encountered before relatively smooth operation of the plant was achieved are enumerated, along with the steps taken to overcome them. In the later runs, both cottonseed and peanut flakes were extracted to a content of 1 percent, or less, of residual oil. Methods are given for the preparation of both cottonseed and peanut flakes for extraction, and the new process for mechanical separation of the pigment components of cottonseed on a pilot-plant scale is described briefly.

292. PROCESSING OF COTTONSEED. III. COLOR DEVELOPMENT IN COTTONSEED OIL DURING STORAGE OF THE SEED AND CRUDE OIL. Williams, P.A.; Boatner, C.H.; Hall, C.M.; O'Connor, R.T.; and Castillon, L.E. J. Am. Oil Chemists' Soc. 24: 362-69. 1947.

Processing cottonseed by the hydraulic press method has been carried out at two mills, one located about 100 miles farther south than the other. The varieties of seed processed and conditions during processing, which included cooking the moistened seed, were essentially the same at both mills. Seeds were also processed at the mill farthest south by the screw-press method for which seed was cooked without added moisture. Oils were stored at different temperatures for 10 months, and samples were periodically refined and bleached. Seeds were also stored at the two mills and periodically processed to permit comparison between the characteristics of oils from stored seed and those of stored oils. The absorption spectra of the crude, refined, and bleached oils were determined. The rate of increase of bleach color in the oils during storage of the seed and crude oils has been correlated with temperature of storage and changes in the absorption spectra of the oils.

280. PROCESSING OF COTTONSEED. II. FACTORS DETERMINING THE DISTRIBUTION AND PROPERTIES OF PIGMENTS IN PRODUCTS PREPARED BY SOLVENT EXTRACTION. Boatner, C.H.; Hall, C.M.; O'Connor, R.T.; Castillon, L.E.; and Curet, M.C. J. Am. Oil Chemists' Soc. 24: 276-83. 1947.

Criteria have been established to provide a basis for obtaining cottonseed oil and meal whose pigmentation may be more or less controlled. For the extraction of oil free of gossypol and gossypurpurin, moisture is excluded from the system and solvents other than water-miscible alcohols and ketones are used. An alternate method is extraction of oil containing essentially all of the pigments of the seed by use of solvents capable of rupturing the glands. The pigment content of the solvent-extracted meals will be determined by the original pigment content of the seed and the solubility of the pigments in the solvent employed. Another method is a special gland flotation process that, in a single operation, gives intact pigment glands and both oil and meal substantially free of pigments.

257. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. IV. PILOT PLANT BATCH EXTRACTION. Pominski, Joseph; Molaison, L. J.; Crovetto, A. J.; Westbrook, R. D.; D'Aquin, E. L.; and Guilbeau, W. F. Oil Mill Gaz. 51(12): 33-39. 1947.

Experience in batch solvent extraction at the Southern Regional Research Laboratory is reported. The portable batch solvent-extraction plant and apparatus used are described and illustrated, and typical extraction data obtained with this unit are tabulated. Batch extraction has been found indispensable for preparing relatively large lots of oils and of defatted cottonseed, peanuts, and other oilseeds for use in pilot-plant work and in chemical, nutritional, protein, and other investigations. The plant has proved a valuable tool for obtaining pertinent processing and development data, including information on equipment and operating methods. An improved portable batch extraction plant will be designed and constructed.

253. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. III. THE EFFECT OF HEAT ON COTTONSEED OIL IN MISCELLAS FROM UNCOOKED AND COOKED FLAKED MEATS. D'Aquin, E. L.; Spardaro, J. J.; Vix, H. L. E.; Pominski, Joseph; Molaison, L. J.; and Pollard, E. F. Oil Mill Gaz. 51(10): 17-19. 1947.

Crude oils were produced on a pilot-plant scale under comparable conditions from a single lot of prime cottonseed by hexane extraction of uncooked and cooked meats and by standard hydraulic pressing of the cooked meats. These oils were compared as to quality. The crude oils were then subjected to various heating temperatures for one hour to simulate evaporative processing in commercial oil and solvent recovery, and the relative effect of heating, as indicated by the color of the oils after caustic refining and bleaching with fuller's earth, was judged. All three oils were of prime grade and were comparable in refining loss, refined color, and bleached color. The oil extracted from the uncooked meats had a lower refining loss and a lighter color than either of the other two oils. In all cases the bleached oils became darker in color as the heating temperature was raised. Color of the oil from uncooked meats appeared to degrade more rapidly at the 150° and 240° F. temperature levels than did that of oil from the cooked meats. Its final bleached color was darker after heating at 240° F. The hydraulic-pressed oil was markedly less affected by heating than either of the hexane-extracted oils and did not exhibit a sharp bleached color degradation as did the latter.

248. PROCESSING OF COTTONSEED. I. PIGMENT DISTRIBUTION IN OILS AND MEALS PRODUCED BY HYDRAULIC AND SCREW PRESS METHODS. Boatner, C. H.; Hall, C. M.; O'Connor, R. T.; Castillon, L. E.; and Curet, M. C. J. Am. Chem. Soc. 24: 97-106. 1947. (Republished: Oil Mill Gaz. 51(11): 46-55. 1947.)

An investigation of the changes that occur in gossypol and other pigments during the cooking and subsequent pressing of cottonseed by the hydraulic-press and continuous screw-press methods was conducted in two parts: (1) laboratory-scale experiments on the effect of cooking and (2) mill-scale experiments on the effect of cooking and pressing. Pigmentation of cooked cottonseed was shown to depend upon the moisture content and period of heating of the seed. The screw-pressed crude oils were more deeply colored than the hydraulic-pressed oils and contained one principal pigment, whereas the latter contained two principal pigments. The absence of significant amounts of gossypol in the crude oils was demonstrated by means of a new technique for the quantitative isolation of gossypol. The crude-oil pigments differed from gossypol, but like gossypol were removed during alkali refining. The pigmentation of the crude oils has been shown to depend principally upon the pigmentation of the original seed and the moisture content of the seed during cooking. On the basis of absorption spectra, it appears that the alkali-refined hydraulic-pressed oils contain two to three pigments originally present in the crude oils whereas the alkali-refined screw-pressed oils contain in addition to these pigments a large number of decomposition products of the principal crude-oil pigment.

224. SOLVENT EXTRACTION AND THE COTTONSEED INDUSTRY. Markley, K. S. Oil Mill Gaz. 51: 16-21. 1946.

The development of solvent methods of processing oilseeds is reviewed and some of the inherent problems are discussed, with numerous references to the literature. Despite the economic advantage of the method in terms of oil recovery, there is in the United States no commercial installation solely devoted to the solvent extraction of cottonseed at present. Solvent extraction of oleaginous materials, patented in England as early as 1856, became established in Europe on a large scale in 1870. Efforts to solvent extract cottonseed in batch operations were made on a large scale as early as 1889. Just before World War II installation of continuous solvent-extraction equipment for soybeans was accelerated in the United States. Work on pigments and pigment glands in cottonseed is described. In conclusion, there is described a unique solvent method developed at the Southern Regional Research Laboratory for processing cottonseed whereby the kernel is separated into intact pigment glands, an oil-solvent mixture, and meal free of oil and of pigment glands.

199. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OIL. EFFECT OF HEAT ON COTTONSEED OIL MISCELLAS. Vix, H. L. E.; Pollard, E. F.; Spadaro, J. J.; and Gastrock, E. A. Ind. Eng. Chem. 38: 635-42. 1946.

This investigation is a practical effort to evaluate the effect of temperature and time of heating during processing operations upon the fixation of objectionable color in miscella obtained by extraction of uncooked cottonseed flakes with commercial hexane. The results reported were obtained from work on one lot of prime cottonseed. Color fixation became objectionable between 150° and 180° and beyond 180° increased rapidly. Properly prepared solvent-extracted cottonseed oils from a prime lot of seed were successfully refined by slightly modified official A. O. C. S. methods, yielding oils of prime quality with low refining losses. The finished oils were equal in quality to high-grade hydraulic-pressed oils. Results have yielded necessary engineering information for design and operation of pilot-plant equipment and for processing operations. Absorption spectra curves ranging through the visible and ultraviolet are reproduced as examples of applicability of this technique to the study of solvent-extracted cottonseed oils. Operating data for heating, as well as complete refining data, are tabulated.

154. SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. BOILING POINT-VAPOR PRESSURE-COMPOSITION RELATIONS FOR MISCELLAS OF OILS IN HEXANE. Pollard, E. F.; Vix, H. L. E.; and Gastrock, E. A. Ind. Eng. Chem. 37: 1022-26. 1945.

The boiling points and densities of mixtures of cottonseed and peanut oils with commercial hexane are reported. They are useful in the design of vacuum evaporators and strippers and for control operations. Boiling-point data are determined at various concentrations of crude cottonseed oil and crude peanut oil over a range of pressures from 160 to 760 mm. absolute in commercial hexane. Observations of the effect of agitation in establishing equilibrium conditions of the oil-solvent mixtures are noted.

151. VISCOSITIES AND DENSITIES OF SOLVENT-VEGETABLE OIL MIXTURES. Magne, F. C.; and Skau, E. L. Ind. Eng. Chem. 37: 1097-1101. 1945.

A pyconometer and a viscometer suitable for use with volatile mixtures and for low-temperature determinations are described. Density and viscosity measurements are made from incipient crystallization to a temperature near boiling point of the solvent for the complete binary systems of cottonseed oil with Skellysolve B (commercial hexane): Cottonseed oil with acetone; cottonseed oil with 2-butanone; peanut oil with Skellysolve B; and soybean oil with Skellysolve B. Density- and viscosity-composition curves for the binary systems are shown and discussed.

2363. RECOVERY OF OIL FROM ACETONE-HEXANE-WATER MIXED SOLVENT EXTRACTS OF RAW COTTONSEED MEATS. King, W. H.; and Frampton, V. L. U. S. Pat. No. 3,025,314, March 13, 1962.

The process for recovering cottonseed oil from a mixed miscella consisting of acetone-hexane-water solvent mixture solutions of cottonseed oil such as are obtained by extracting raw cottonseed meats by the prodecure described in related patent No. 2,950,158.

1705. DOUBLE SOAK FILTRATION-EXTRACTION OF VEGETABLE OIL-BEARING MATERIALS. D'Aquin, E. L.; Vix, H. L. E.; Graci, A. V., Jr.; Gastrock, E. A.; and Spadaro, J. J. U. S. Pat. No. 2,857,411, October 21, 1958.

An improved method of solvent extraction applicable to vegetable oil-bearing materials. Exceptionally low solvent-to-meal ratios are possible by use of the patented process.

1633. PRETREATMENT OF OILSEED MEATS. King, W. H. U. S. Pat. No. 2,820,047, January 14, 1958.

A process for producing oil from vegetable oilseeds, which involves the pretreating of the oilseed material with little or no heat.

1483. IMPROVING COTTONSEED OIL WHILE PREPARING THE SEED FOR OIL EXTRACTION. King, W. H.; and Thurber, F. H. U. S. Pat. No. 2,784,205, March 5, 1957.

This invention relates to a process for improving the color stability characteristics of cottonseed oil. More particularly, the invention provides a process of treating cottonseed meats containing free gossypol while they are being prepared for oil extraction, to produce prepared meats that yield cottonseed oils having little tendency to undergo color reversion.

1327. CENTRIFUGAL EXTRACTOR. Holzenthal, L. L. U. S. Pat. No. 2,734,635, February 14, 1956.

An apparatus for continuously mixing granular solid materials, such as oilseed meats, with liquids such as oil solvents and separating the resultant solution, comprising a porous tube into which the materials, slurried together, are placed, centrifugally separated, and recombined and reseparated as many times as desired.

1322. MEAL RECYCLING SOLVENT EXTRACTION PROCESS. Pominski, Joseph; and Vix, H. L. E. U. S. Pat. No. 2,729,661, January 3, 1956.

A process that provides a method of recycling desolventized and dried solvent-extracted meals to improve the filtration characteristics of oil-containing particles of vegetable oil-bearing materials.

1256. COTTONSEED OIL EXTRACTION PROCESS. Gastrock, E. A.; Vix, H. L. E.; D'Aquin, E. L.; Spadaro, J. J.; and Graci, A. V., Jr. U. S. Pat. No. 2,726,253, December 6, 1955.

A process for solvent extraction of cottonseed oil from cottonseed, comprising mildly cooking cottonseed meat flakes while controlling their moisture content so that in the early stages of the cooking the meats contain between 14 to 26 percent moisture and in the final stages 6 to 12 percent moisture, using an overall cooking time of 30 to 70 minutes and increasing the cooking temperature from an initial 170 to 210° F. to a final temperature near 235° F.; crisping the

cooked meats by reducing the temperature below 130° F. and the moisture content by 2 to 4 percent; passing the particles between flaking rolls adjusted to compress the particle masses to a thickness of 0.008 to 0.012 in.; and then mixing the prepared cottonseed meat particles with an oilseed extraction solvent, subjecting the resultant slurry to filtration, repeating the mixing with the solvent and the filtering a plurality of times.

1255. TREATING VEGETABLE OIL-BEARING MATERIALS TO OBTAIN MEALS OF IMPROVED NUTRITIVE VALUE. King, W. H.; Thurber, F. H.; and Altschul, A. M. U. S. Pat. No. 2,726,155, December 6, 1955.

A process comprising adjusting the moisture content of soybean or cottonseed meats to 20 to 50 percent and adjusting the pH of the meats to a value between 4 and 9.5 and which differs from the natural pH of the materials by at least 0.5 pH units; dehydratively heating the moisture- and pH-adjusted meats at 60 to 225° F. for 20 to 40 minutes until their moisture content is reduced to about 7 to 13 percent, with the temperature being kept below about 200° F. for 10 to 20 minutes.

OIL - PROCESSING

2675. PHYSICAL AND CHEMICAL PROPERTIES OF ALUMINA-BLEACHED COTTONSEED OIL. Kuck, J. C.; Pons, W. A., Jr.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 41: 101-104. 1964.

No detectable amount of polymerization or triene conjugation occurred on bleaching refined cottonseed oil with either activated alumina or sulfurous acid-treated alumina, although insignificant amounts of diene conjugation and trans-isomerization occurred. AOM stability, tocopherol content, and fatty acid composition, as determined by gas-liquid chromatography (GLC), were comparable with values obtained with oil bleached with natural earth. Hydrogenation of the oils proceeded normally. Taste panel evaluations of the deodorized oils revealed their flavor stability to be equal to that of the same oil bleached with AOCS official earth.

2466. BLEACHING OF REFINED COTTONSEED OIL WITH MODIFIED ALUMINA ADSORBENTS. Pons, W. A., Jr.; Kuck, J. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 40: 10-13. 1963.

Activated aluminas, modified by treatment with inorganic and organic acids, were tested for their efficiency as bleaching agents and for their ability to eliminate the response of refined cottonseed oil to the Halphen test. The best of these adsorbents, an alumina containing catalytic amounts of adsorbed sulfurous acid, is a bleaching agent that is effective in removing problem pigments from off-colored cottonseed oils and is superior to activated aluminas in removing green chlorophyll-like pigments. Oils bleached with the catalytic adsorbent are negative to the Halphen test and are free of sulfur. Study of the rate of reduction in the Halphen test response as a function of bleaching time and temperature indicated that sulfurous acid treated alumina has a pronounced catalytic effect. The Halphen test response is eliminated during a 30-minute bleaching period at 225° C. Deodorization conditions employed had little effect on Halphen test reduction.

2285. REACTIVATION OF ALUMINA USED IN BLEACHING OF OFF-COLORED COTTONSEED OILS. Kuck, J. C.; Pons, W. A., Jr.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 39: 84-86. 1962.

Spent alumina recovered from bleaching cottonseed oil with activated alumina can be reactivated by simple incineration at 400 to 700° C. and remoistening to at least 10 percent moisture content. The cycle of bleaching and regeneration may be repeated indefinitely with only nominal mechanical losses of alumina. Losses of refined oil by entrainment in activated alumina need be only 0.5 percent.

2201. EFFECT OF EXTRACTION TEMPERATURE AND REFINING ON THE HALPHEN-TEST RESPONSE OF COTTONSEED OIL. Bailey, A. V.; Magne, F. C.; Pittman, R. A.; and Skau, E. L. J. Am. Oil Chemists' Soc. 38: 505-06. 1961.

Cottonseed oil of higher Halphen-acid moiety concentration can be obtained by hot hexane extraction of cottonseed meats from which the oil has already been partially extracted at room temperature. The intensity of the Halphen-test color solution is affected by many nitrogen-containing substances, including soybean phosphatides.

2200. PROPERTIES OF OIL EXTRACTED FROM COTTONSEED WITH ACETONE-HEXANE-WATER SOLVENT MIXTURE. King, W. H.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 38: 497-99. 1961.

Slightly more neutral oil was obtained on the exhaustive extraction of raw cottonseed meats with the acetone-hexane-water (AHW) solvent mixture than was obtained under the same conditions with commercial hexane. Although most of the gossypol originally present in the seed is extracted with the oil when the AHW solvent mixture is used, the crude oils refined and bleached to yield oils of excellent colors. Methods of recovery of the solvent from the mixed solvent miscellas are reported, together with the refining and bleaching data for the recovered oils.

2075. BLEACHING OF OFF-COLORED COTTONSEED OILS. Pons, W. A., Jr.; Kuck, J. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 38: 104-07. 1961.

The adsorption of fixed red pigments from refined off-colored cottonseed oil by several adsorbents is described by the empirical Freundlich adsorption isotherm. Numerical values for the coefficient and the exponent in the Freundlich equation were determined for several oils and several adsorbents. Activated alumina was found to be a superior adsorbent for removing the red color bodies. The variations from oil to oil in the numerical values of the coefficient and the exponent of the Freundlich equation were smaller for the several aluminas than they were for the other adsorbents studies. The particle size and moisture content of the alumina and the temperature of activated adsorption are important in determining the effectiveness with which the red color bodies are removed from the oils. The conditions required for optimum bleaching with alumina had no detectably adverse effect on oil quality.

1690. POSITIONAL ISOMERS FORMED DURING THE HYDROGENATION OF COTTONSEED OIL. Chahine, M. H.; Cousins, E. R.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 35: 396-401. 1958.

A commercial cottonseed oil was hydrogenated under nonselective, normal, and selective conditions. The operating variables used were within the ranges of those ordinarily found in large-scale operations. For each run, samples were withdrawn at iodine values of approximately 75, 62, and 48; and these samples were analyzed for the position of the double bonds, content of trans isomers, and content of linoleins. Double bonds were found in the 6 through 14 positions of the monounsaturated fatty acid groups resulting from the hydrogenations. On the basis of the percentage distribution of the double bonds, there appeared to be no marked

tendency for the linoleoyl group to form 9- or 12-isomers of the oleoyl group. In the early stages of the selective hydrogenation, the rate at which double bonds shifted from the 9-position was greater than the rate at which double bonds were hydrogenated. The conditions of hydrogenation did not have a marked effect on the distribution of the double bonds at iodine values of about 62 and 48. The conditions of hydrogenation did have marked effect on the percentage of trans bonds. At an iodine value of approximately 75, the content of trans bonds, expressed as weight percentage of trielaidin, was 9.4 for the nonselective hydrogenation and 27.3 for the selective hydrogenation; at an iodine value of approximately 48, these values increased to 21.6 and 37.7, respectively.

1687. NOTE ON THE IMPROVEMENT OF COLOR IN OFF-COLORED COTTONSEED OILS. Frampton, V. L.; Kuck, J. C.; Dechary, J. M.; and Altschul, A. M. J. Am. Oil Chemists' Soc. 35: 18, 20. 1958.

The use of polyfunctional aliphatic amines in conjunction with the standard refining and bleaching operations induces a marked improvement in the color of typically off-colored and unbleachable cottonseed oils.

1544. THE VALUE OF COTTONSEED OILS AS AFFECTED BY THE DEGREE OF OIL EXTRACTION AND THE METHOD OF MEATS PREPARATION. Eaves, P. H.; Spadaro, J. J.; Crovetto, A. J.; Gastrock, E. A.; and D'Aquin, E. L. Oil Mill Gaz. 62(1): 9-13. 1957. (Republished Oil Mill Gaz. 64(6): 7-11. 1959.)

Data are reported showing that the value of a crude cottonseed oil as determined by the present trading rules is affected by the degree of extraction, the method of preparing the meats for extraction, and the type of seed used. Seed of low and high oil content were evaluated. The data reported are intended for use as a guide since conditions such as seed variety, seed quality, type of processing used, desired plant capacity, and operating control will vary with each mill and can affect the economically optimum degree of extraction for the individual mill.

1536. NOTE ON GOSSYPOL AND ITS RELATION TO COLOR FIXATION IN COTTONSEED OIL. Berardi, L. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 34: 399-401. 1957.

The crude oils studied contained from 0.00 to 0.04 percent gossypol. Much of the gossypol added to crude cottonseed oils disappeared in one hour. The disappearance of gossypol from refined, bleached, and deodorized cottonseed oil, from highly purified tripelargonin, and from ethyl acetate is demonstrable after the lapse of one hour. It is suggested that the initial reaction of gossypol in the oils is an ester exchange reaction. It is further suggested that secondary reactions result in the production of a red coloration product that is not removable from the oil by the standard refining and bleaching methods.

1393. CONSISTENCY OF MIXTURES OF COTTONSEED AND PARAGUAYAN PALM KERNEL OILS. Landmann, Werner; and Feuge, R. O. J. Am. Oil Chemists' Soc. 33: 308-11. 1956.

Mbocaya oil, the kernel oil from the Paraguayan palm, Acrocomia Totai Mart, is available in Paraguay as an edible oil. It has an iodine value of about 30, which is three times that of ordinary commercial palm kernel oils, but its point of complete melting is about 21° C. For technical and economic reasons, shortening-like products containing the oil are best prepared by blending with a hydrogenated vegetable oil, such as cottonseed oil. Various blends of hydrogenated cottonseed oil and palm kernel oil were prepared and their consistency was measured over a temperature range of 10° to 40° C. Certain mixtures yielded desirable consistency curves and possessed other good characteristics. Interesterification of

hydrogenated cottonseed oil and palm kernel oil to produce shortening-like products was investigated, as well as the use of hydrogenated palm kernel oil in the mixtures.

1383. REFINING COTTONSEED OIL AT HIGH RATES OF SHEAR. Feuge, R. O.; Vicknair, E. J.; and Lovegren, N. V. J. Am. Oil Chemists' Soc. 33: 344-47. 1956.

Use of extremely high rates of shear during the caustic soda refining of crude cottonseed oils was investigated. The effects of temperature, strength of the caustic soda solution, amount of excess caustic soda, and duration of mixing on the color of the refined oil were determined in order to establish the conditions most desirable for high-shear refining. In every test the refined oils and the refined and bleached oils were lighter in color when high-shear agitation was used instead of conventional agitation. The improvement in color was not always marked. Some oils did not refine to a satisfactory color by either the high-shear or conventional method. However, other crude oils that yielded dark oils on conventional refining yielded much lighter oil after high-shear refining. Absorption spectra in the visible region were obtained for several crude, refined, and refined and bleached oils, and an indication of the effect of high-shear agitation on the various types of color bodies present in crude cottonseed oil was established.

1377. THE DIFFICULTLY EXTRACTABLE LIPIDS OF COTTONSEED MEATS, THEIR COMPOSITION AND EFFECT ON THE REFINING CHARACTERISTICS OF THE CRUDE OILS. Eaves, P. H.; Spadaro, J. J.; D'Aquin, E. L.; Crovetto, A. J.; Cirino, V. O.; Stansbury, M. F.; and Hoffpauir, C. L. J. Am. Oil Chemists' Soc. 33: 639-45. 1956.

Crude lipid fractions were obtained from raw, tempered, and cooked cottonseed meats in the order of their difficulty of extraction, and their proximate composition was determined. Composition varied with the degree of exhaustiveness of extraction. The fractions obtained by more exhaustive extraction contained greater amounts of undesirable nonneutral oil and lesser amounts of the desirable neutral oil. The lipid fractions from raw and tempered meats contained large amounts of impurities, whereas the crude lipid fractions from cooked meats were relatively low in impurities. Crude oils equivalent to varying degrees of total lipids extraction were reconstituted from the crude lipid fractions and evaluated for refining characteristics. The impurities content of the reconstituted oils varied as the degree of total lipids extraction, and such increases were generally reflected in disproportionate increases in refining losses or oil color or both.

956. IMPROVED APPARATUS AND METHODS FOR QUANTITATIVE HYDROGENATION. Pack, F. C.; and Planck, R. W. J. Am. Oil Chemists' Soc. 30: 461-63. 1953.

Improved apparatus and techniques for the measurement of olefinic unsaturation by quantitative hydrogenation are described. These involve use of a special sample-introducing device, of n-alkyl esters of monobasic fatty acids as solvents, and of palladium carbon catalysts, thereby speeding up the determinations, improving their precision, and extending their utility.

846. NEWLY DEVELOPED PILOT-PLANT BATCH DEODORIZER HAS MANY USES IN VEGETABLE OIL RESEARCH. Wellborn, W. A.; Molaison, H. J.; and D'Aquin, E. L. Food Eng. 24(10): 171. 1952.

A batch deodorizer with a working capacity of 300 to 600 lbs. can be operated at temperatures up to 450° F., at a vacuum as high as 29.7 in., and with various amounts and pressures of blowing steam. The unit is 6 ft. 2 in. high by 30 in. inside diameter. The shell is constructed of 3/8-in. mild steel.

816. THE CONIDENDROLS AS INHIBITORS OF OXIDATION AND POLYMERIZATION. Mack, C. H.; and Bickford, W. G. J. Am. Oil Chemists' Soc. 29: 428-30. 1952.

Alpha- and beta-conidendrol have been compared with accepted stabilizers as antioxidants to prevent rancidity in fats, oils, and fat-containing candies; as stabilizers against aging and resinification in GR-S-type polymers; and as additives for inhibiting oxidation and polymerization of vinyl-type monomers. The conidendrols were approximately equal or superior to other stabilizers in current use in their respective fields.

813. PHASE RELATIONS IN THE SOLVENT WINTERIZATION OF COTTONSEED OIL IN 85-15 ACETONE-HEXANE MIXTURE AT REDUCED HOLDING-TIMES. Boucher, R. E.; and Skau, E. L. J. Am. Oil Chemists' Soc. 29: 455-56. 1952.

Systematic winterization data were obtained on a laboratory scale for different oil-solvent ratios and temperatures using a technique previously described. The results indicated that under the static chilling conditions used, the holding time can be reduced from 3 hours to 1 hour by using either a higher oil concentration, a lower chilling temperature, or both.

812. PHASE RELATIONS IN THE SOLVENT WINTERIZATION OF MOLECULARLY REARRANGED PEANUT OIL AND COTTONSEED OIL. Boucher, R. E.; and Skau, E. L. J. Am. Oil Chemists' Soc. 29: 382-85. 1952.

Systematic phase-relation data, obtained to determine the effect of either mild or extensive molecular rearrangement on the winterization behavior of peanut or cottonseed oils, show that if the molecular rearrangement step is introduced before solvent winterization, larger percentages of solid must be removed to obtain a winterized oil; lower yields result; lower temperature and longer chilling periods are required; and the settling qualities of the solid separating are markedly impaired.

741. A COMPARATIVE EVALUATION OF SEVERAL ANTIOXIDANTS IN EDIBLE FATS. Moore, R. N.; and Bickford, W. G. J. Am. Oil Chemists' Soc. 29: 1-4. 1952.

Thirteen compounds have been compared with respect to their antioxidant activity in concentrations of 0.01, 0.05, and 0.10 percent in edible cottonseed oil, the same oil hydrogenated to shortening consistency, and in lard that is essentially free of naturally occurring antioxidants. None of the compounds exhibited significant antioxidant activity in the cottonseed oils, when used in a concentration of 0.01 percent, but they were effective in stabilizing lard under these conditions. Propyl gallate was the most effective of the compounds tested for the vegetable fats.

738. EFFECT OF WINTERIZATION ON THE COMPOSITION AND STABILITY OF COTTONSEED OIL. Mack, C. H.; Moore, R. N.; and Bickford, W. G. J. Am. Oil Chemists' Soc. 29: 14-16. 1952.

The relations among tocopherol content, fatty acid composition, and autoxidative stability in a commercially refined and bleached cottonseed oil, in the same oil after winterization, and in the corresponding stearine press cake were investigated. Unsaturation of the glycerides increased accompanied by an increase in the tocopherol content of the winterized over the unwinterized oil. The reverse was true of the stearine press cake.

736. STABILITY OF COTTONSEED AND PEANUT OILS TO AUTOXIDATION. Dollear, F. G. Potato Chipper 11(11): 24, 26, 28, 30, 32. 1952.

The influence of the type and proportion of the various fatty acid components of the glycerides and the presence of natural or added antioxidants, or pro-oxidants, and of synergists or metal deactivators or both on the resistance of cottonseed and peanut oils to autoxidation has been studied. The stability of these oils can be increased by hydrogenation or by the addition of some types of antioxidants.

731. RE-REFINING COTTONSEED OIL AT HIGH RATES OF SHEAR. Kroonen, J. G.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 29(2): 65-71. 1952.

In re-refining cottonseed oils at high rates of agitation and shear, color decreased as the rate of agitation increased until a rate was reached beyond which greater agitation had little effect. About 0.1 percent of sodium hydroxide was the minimum and 0.2 percent the optimum amount required to produce the maximum improvement in color at high rates of shear. With higher temperatures during re-refining, color decreased; between 15° and 65° C., the removal of color was almost a linear function of the re-refining temperature.

721. INFLUENCE OF CONDITION OF SEED ON THE SPECTRAL PROPERTIES OF CRUDE AND PROCESSED HYDRAULIC COTTONSEED OILS. O'Connor, R. T.; Pominski, C. H.; Heinzelman, D. C.; Howell, Hilda; and Von Der Haar, Patricia. J. Am. Oil Chemists' Soc. 29(6): 220-24. 1952.

Ultraviolet and visible spectra of seven crude hot- and cold-pressed hydraulic cottonseed oils and of the refined, bleached, and deodorized oils processed from them are illustrated and discussed. Diene conjugation was found generally to be greater in oils obtained by hot than by cold hydraulic pressing and in oils from seed stored for some time. Individual gossypol pigments varied more in character in hot-pressed than in cold-pressed oils. The spectrum of carotenes was masked in crude oils, especially in hot-pressed ones, but was apparent in refined oils. The carotenes were removed by bleaching. The deep red color, especially of oils from stored seed, is shown to be due to unidentified chromophores rather than to gossypurpurin. Absorption in the red region of the spectrum is attributed to pheophytin.

651. PREPARATION OF A RELATIVELY ANTIOXIDANT-FREE VEGETABLE OIL. Fisher, G. S.; and Bickford, W. G. Am. J. Pharm. 123(7): 233-36. 1951.

A method for preparing essentially antioxidant-free and nearly colorless vegetable oils is described. The oil is dissolved in pentane and the solution is passed through a specially prepared adsorption column containing layers of activated carbon and filter aid, activated, alumina, and anhydrous sodium sulfate. Peanut oil treated by this method yielded a product having a keeping time of about 2 hours by the active oxygen method at 97.7° C. Such substrate oils are useful in testing and distinguishing between antioxidants and synergists and in preparing vitamin-free fat diets.

649. BLEACHING OF COTTONSEED OIL IN HEXANE. Feuge, R. O.; and Janssen, H. J. J. Am. Oil Chemists' Soc. 28: 429-32. 1951.

Cottonseed oil dissolved in commercial hexane was bleached readily by activated clay and carbon, and the reduction in color was greater than that obtained in the absence of a solvent, other conditions being similar. Countercurrent bleaching of cottonseed oil-hexane solutions in columns packed with adsorbents proved to be highly efficient with respect to the utilization of the adsorptive capacity of a given quantity of clay and also with respect to the amount of oil lost by adsorption. Columns of spent bleaching clay could be regenerated easily with acetone. The Lovibond red color of the first batch of oil bleached in one such column was 2.8, whereas that of the 20th batch, obtained after the column had been used and regenerated 19 times, was 3.8.

646. PHASE RELATIONS PERTAINING TO THE SOLVENT WINTERIZATION OF COTTONSEED OIL IN HEXANE AND IN ACETONE-HEXANE MIXTURE. Boucher, R. E.; and Skau, E. L. J. Am. Oil Chemists' Soc. 28: 483-87. 1951.

Systematic phase relation data pertaining to the solvent winterization behavior of a refined cottonseed oil have been obtained for commercial hexane and a mixed solvent consisting of 85 percent by weight of acetone and 15 percent of hexane. The effects of oil-solvent ratio, chilling temperature, holding time, and agitation on the percentage of solid removed, the degree of

winterization, and the settling qualities of the solid separating are shown in graphs. These data, with those previously reported for acetone (J. Am. Oil Chemists' Soc. 27: 556-64. 1950), afford a basis for the selection of the optimum conditions and procedures in the application of solvent winterization to cottonseed oil.

590. AUTOXIDATION OF FATS AND OILS. Swift, C. E.; and Dollear, F. G. Yearbook Agr., U. S. Dept. Agr. 1950-51: 544-50.

Autoxidation of fats and oils is the action that takes place when these products are exposed to air. Autoxidation causes rancidity and deterioration in other ways and is the cause of serious economic losses. Radical discoveries that have been made recently concerning some of the basic reactions involved in this phenomenon are discussed as contrasted with earlier views. Some substances that tend to prevent autoxidation, called antioxidants, occur naturally in fats, and others of natural or synthetic origin may be added to fats to inhibit autoxidation. To a limited extent, certain antioxidants are utilized by industry under Federally specified regulations. Hardening fats by hydrogenation produces considerably increased stability in the hardened as compared to the unhardened oil. Empirical methods of preventing autoxidation provide limited effectiveness, but more recent fundamental knowledge offers a basis for developing completely adequate solutions to the problems of autoxidations.

587. OIL PROCESSING THROUGH THE AGES. Markley, K. S. Yearbook Agr., U. S. Dept. Agr., 1950-51: 497-503.

Modern developments that have led to increased recovery of crude oils from various oleaginous raw materials and to increased yields and improved quality of derived finished products are reviewed. The review includes discussion of the development and application of continuous screw pressing, solvent-extraction, continuous centrifugal refining, methods of bleaching and deodorization, and application of hydrogenation to the conversion of liquid oils to plastic and solid fats. The review also includes discussion of methods of converting semidrying to drying oils and recovery of byproducts, such as still pitch, fatty acids, phosphatides, vitamin concentrates, and sterols. There is brief reference to the development of modern detergents, wetting and emulsifying agents, modern decorative and protective coatings, plastics, and other products.

583. VEGETABLE OILS AND FATS FOR EDIBLE USE. Feuge, R. O. Yearbook Agr., U. S. Dept. Agr. 1950-51: 520-28.

Refining, bleaching, winterization, hydrogenation, and deodorization, which are the more or less standard unit processes used in the conversion of crude vegetable fats and oils into various edible products, are described in detail in the order in which they are customarily applied. Details given include the description of equipment and materials, the conditions used industrially, and an explanation of the chemistry or physics involved in each process.

549. VISCOSITIES OF COTTONSEED AND PEANUT OIL-HEXANE MISCELLAS IN ENGLISH UNITS. Decossas, K. M.; Deckbar, F. A., Jr.; and Hecker, J. L. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-304. 1951. (Processed.) (See also 492.)

Constant composition curves of viscosity versus temperature for refined and winterized cottonseed oil-commercial hexane miscellas and for like miscellas with peanut oil are presented. Tables of viscosities of these vegetable oil miscellas at various temperatures and composition (2) were converted from data in metric units to English units and plotted as intermediate viscosity-composition isotherms. Readings of viscosity versus temperature taken off these intermediate curves were plotted. Absolute viscosity, mu, as ordinate expressed in pounds per foot-hour, and temperature, t, as abscissa expressed in degrees Fahrenheit were plotted. These graphs are complementary to others in English units on boiling points, densities, and gravities of cottonseed and peanut oil miscellas.

522. IMPROVED PROCEDURE FOR CLEANING GLASSWARE USED IN DETERMINING THE STABILITY OF FATS AND OILS BY THE ACTIVE OXYGEN METHOD. Fore, S. P.; Moore, R. N.; and Bickford, W. G. J. Am. Oil Chemists' Soc. 28: 73-74. 1951.

A method is described for cleaning the glassware required in using the active oxygen method for determining the stability of fats and oils. The method, which uses a synthetic detergent instead of hot or cold chromic-sulfuric acid, leads to improved reproducibility of the determinations. The method described has given reliable results in use by the authors almost daily for more than a year.

502. PHASE RELATIONS PERTAINING TO THE SOLVENT WINTERIZATION OF COTTONSEED AND PEANUT OILS IN ACETONE. Skau, E. L.; Dopp, W. N.; Burleigh, E. G.; and Banowetz, L. F. J. Am. Oil Chemists' Soc. 27: 556-64. 1950.

Systematic physical-chemical data on the solvent-winterization behavior of cottonseed and peanut oils with acetone have been obtained that should serve as a basis for selecting the conditions necessary for the effective solvent winterization of these oils in acetone. The two oils are only partly miscible with acetone below certain temperatures that have been determined. In peanut oil this phenomenon may interfere with the winterization process within a certain range of concentrations, but in cottonseed oil the separation into two liquid phases does not occur until some 5° C. below the temperature required for adequate winterization. Complete data for a 3-hour holding time have been obtained for three cottonseed oils ranging in iodine value from 106.1 to 116.4. Tables and graphs have been constructed to show the effect of oil-solvent ratio, chilling temperature, holding time, agitation, and iodine value of the original oil on the percentage of solid removed and on the degree of winterization and iodine value of the winterized oil. Similar data have been obtained for a refined peanut oil insofar as possible without interference from separation into two liquid phases. It seems probable that if acetone were used as the winterization solvent for peanut oil, the separation into two liquid layers and the sensitivity of this phenomenon to moisture might be a source of processing difficulties, especially if filtration instead of centrifugation were used to separate the solid from the supernatant.

497. DENSITY-COMPOSITION DATA FOR COTTONSEED OIL-SOLVENT MIXTURES. Magne, F. C.; Hughes, E. J.; and Skau, E. L. J. Am. Oil Chemists' Soc. 27: 552-55. 1950.

Density-composition-temperature data are reported for binary systems of a refined cottonseed oil with methyl pentane, diethyl ether, trichloroethylene, and tetrachloroethylene over a wide temperature range--from the point of incipient phase separation to within 10° to 15° C. of the boiling point of the solvent. These data can be used to calculate the density knowing the composition and temperature or, conversely, the composition knowing the density and the temperature. An equation has been derived that makes it possible to adjust these data so that they apply with reasonable accuracy to other refined and crude cottonseed oils and even to soybean or peanut oil systems with the same solvents.

492. DENSITIES AND GRAVITIES OF COTTONSEED AND PEANUT OIL MISCELLAS IN ENGLISH UNITS. Decossas, K. M.; Deckbar, F. A., Jr.; and Hecker, J. L. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-292. (Processed.) 1950.

Data in the literature on density-temperature-constant composition parameters for refined and winterized cottonseed oil-commercial hexane miscellas and for refined winterized peanut oil-commercial hexane miscellas are replotted in English units; namely, densities in pounds per cubic foot and per gallon, and temperatures in degrees Fahrenheit. The specific gravities are

calculated on a basis of the density of water at 60° F. These plots complement those previously published in English units on boiling points of cottonseed and peanut oil miscellas. Replotting these data will render them more useful and suitable for design calculations, plant operations, and interpretations of operating data.

447. BOILING POINTS OF COTTONSEED AND PEANUT OIL MISCELLAS IN ENGLISH UNITS. Decossas, K. M.; Mackey, H. A.; and Heughan, G. P. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-257. (Processed.) 1950.

Data previously obtained on boiling point-vapor pressure-constant composition parameters for crude cottonseed oil-commercial hexane miscellas and crude peanut oil-commercial hexane miscellas have been replotted in English units; namely, in inches of mercury vacuum and in degrees of temperature Fahrenheit. Constant composition parameters are at 0, 50, 60, 70, 80, 85, 90, 93, 95, 97, and 98 percent oil by weight. The purpose of replotting is to render these data more useful and suitable for design calculations, plant operations, and interpretations of operating data.

422. THE INFLUENCE OF PROCESSING ON THE SPECTRAL PROPERTIES OF VEGETABLE OILS. O'Connor, R. T.; Field, E. T.; Jefferson, M. E.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 26: 710-18. 1949.

Data are presented on the results of a detailed examination of the absorption spectra from 200 mμ to 720 mμ of typical cottonseed, soybean, peanut, sesame, okra seed, and rice bran oils. The absorption measurements were made on both crude and processed oils at successive processing steps of refining, bleaching, and deodorizing. Findings are summarized for the ultraviolet region from 200 mμ for the near ultraviolet and the blue to blue-green portions of the visible region between 320 mμ and 500 mμ, and for the visible portion of the spectra above 500 mμ.

283. NORCONDIDENDRIN: A NEW ANTIOXIDANT FOR FATS AND OILS. Fisher, G. S.; Kyame, Lillian; and Bickford, W. G. J. Am. Oil Chemists' Soc. 24: 340-43. 1947.

Norconidendrin, prepared by hydrolysis of conidendrin, a polyphenolic substance obtained by extraction of sulfite waste liquors from the manufacture of pulp from western hemlock, possesses antioxidant activity in hydrogenated and unhydrogenated cottonseed and peanut oils, in a peanut oil essentially free of natural antioxidants, and in lard. Norconidendrin, as well as other polyphenols tested, exhibited greater antioxidant activity in the peanut oils used than in several other vegetable oils and still greater activity in substrates which contained only small amounts of natural antioxidants. Effectiveness was appreciably enhanced by the addition of acid-type synergists. Norconidendrin may be added to an oil before deodorization without affecting odor, color, or flavor.

282. CORRELATION OF STABILITY WITH FATTY ACID COMPOSITION OF HYDROGENATED VEGETABLE OIL. Fisher, G. S.; Bickford, W. G.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 24: 379-82. 1947.

Cottonseed, peanut, and linseed oils were hydrogenated under selective and nonselective conditions and samples withdrawn periodically for determination of their fatty acid composition, keeping quality, and other characteristics. Graphical and statistical analysis of the results indicate that when cottonseed or peanut oil is hydrogenated under either selective or nonselective conditions, the change in the reciprocal of the keeping quality, as measured by the active oxygen method, is proportional to the change in linolein content up to the point of disappearance of linolein. After all linoleic acid has disappeared, the change in keeping quality is proportional to the change in olein content. When linseed oil is hydrogenated under selective conditions, the change in the reciprocal of the keeping quality is proportional to the change in the linolenin content up to the point of disappearance of the linolenin.

192. MODIFICATION OF VEGETABLE OILS. V. RELATIVE REACTIVITIES TOWARD HYDROGENATION OF THE MONO-, DI-, and TRIETHANOID ACIDS IN CERTAIN OILS. Bailey, A. E.; and Fisher, G. S. Oil & Soap 23: 14-18. 1946.

From the analysis of several series of hydrogenated cottonseed, soybean, and linseed oils, estimates have been made of the relative readiness of hydrogenation of oleic, iso-oleic, linoleic, and linolenic acids, and an isomer of linoleic acid which presumably had its double bonds in the 9:10 and 15:16 positions. Under selective conditions of hydrogentation, the relative reactivities of the unsaturated fatty acids toward hydrogen may be approximately represented by the following whole numbers: oleic acid 1; iso-oleic acid 1; isolinoleic acid 3; linoleic acid 20; and linolenic acid 40. Use of "nonselective" conditions reduces the reactivity of linoleic and linolenic acids as a group in comparison with that of oleic and isolinoleic acids as a group but does not appreciably alter the relative reactivities of the two acids within the same group. In nonselective hydrogenation, the ratio of reactivity of linoleic acid to that of oleic acid may be as low as 5, as compared with 20 or above in selective hydrogenation. The hydrogenation of linoleic acid to stearic acid appears to proceed substantially in two stages, with intermediate desorption of oleic acid from the catalyst, but there is evidence that the hydrogenation of linolenic acid may in part proceed directly to oleic acid, without intermediate desorption of linoleic acid. In both selective and nonselective hydrogenation, the hydrogenation of linolenic acid to linoleic acid and to isolinoleic acid appears to proceed at approximately equal rates.

180. A PREPARATION OF LEAD THIOCYANATE. Lambou, M. G.; and Dollear, F. G. Oil & Soap 23: 97-101. 1946.

A comprehensive investigation of previously published methods is reported and lead thiocyanate made by these methods is compared with that made by an improved method. All products were assayed for purity and tests were made of their capacity for saturating double bonds in cottonseed, peanut, and soybean oils. The lead thiocyanate prepared according to the recommended procedure was found superior in stability, purity, and ability to produce dependable thiocyanogen values.

160. FLAVOR REVERSION IN EDIBLE FATS. Bailey, A. E. Quartermaster Corps Manual, QMC 17-7. 1945. (Republished: Oil & Soap 23: 55-88; also Oil Mill Gaz. 50(12): 46-50. 1946.)

Certain fats which develop objectionable flavor with extremely slight oxidation, including particularly fish and vegetable oils containing linolenic acid, are discussed. The flavor reversion of both unhydrogenated and hydrogenated oils is considered. Flavor reversion in hydrogenated oils appears definitely associated with the presence of highly unsaturated acids. In contrast, there is some evidence to indicate that reverted flavor in hydrogenated oil may be simply a product of the glycerides of the oil.

157. UNITED STATES MANUFACTURERS OF EQUIPMENT FOR PROCESSING COTTONSEED AND PEANUTS INTO OILS, MEAL, AND BYPRODUCTS. Anonymous. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-98. 1945. (Processed.)

This is an alphabetical list of most of the manufacturing companies serving processors of cottonseed and peanuts. In addition a cross listing is given under the type of equipment manufactured.

75. LIST OF ANTIOXIDANTS PROPOSED FOR THE PRESERVATION OF EDIBLE FATS (WITH A SELECTED BIBLIOGRAPHY OF PAPERS AND PATENTS). Anon. U. S. Dept Agr., Bur. Agr. Ind. Chem. AIC-54. 8 pp. (Processed). 1948.

A list of 38 compounds, or groups of compounds, which may be used as antioxidants for edible fats is given, followed by a partial list of sources of supply. A bibliography of 72 references is given, keyed by number to the compounds to which they are applicable.

68. MOLECULAR DISTILLATION OF A HYDROGENATED COTTONSEED OIL AND CERTAIN CHARACTERISTICS OF THE DISTILLED FRACTIONS. Singleton, W. S.; and Bailey, A. E. Oil & Soap 21(6): 157-59. 1944.

Fractionation of a molecularly distilled oil occurs on the basis of variations in molecular weight of the glycerides. The composition of cottonseed oil is such that there is considerable separation of the glycerides according to their degree of unsaturation. Molecular distillation causes segregation of tocopherols and related compounds similar to that observed in peanut oil. However, the fractions first distilled from the oil are relatively weak in antioxygenic properties. The tocopherols in the potent fractions aremore effective than alpha-, but less effective than gamma-tocopherol.

66. VISCOSITIES AND DENSITIES OF HYDROGENATED COTTONSEED OILS. Wakeham, Helmut; and Magne, F. C. Ind. Eng. Chem. 36: 568-70. 1944.

Studies have been made on the viscosities and densities of cottonseed oils from various sources as related to iodine number and temperature. Properties of hydrogenated cottonseed oils prepared in the laboratory from a commercial refined and deodorized oil are compared with those of commercially refined unhydrogenated oils from three different sources. These samples were supplemented with commercially hydrogenated oils of routine batches obtained from a local processor. In addition to iodine number and temperature, the properties of melting range, solidification range, refractive index, and free fatty acid calculated as oleic are reported.

62. EFFECT OF METALLIC SALTS ON THE STABILITY OF PALM AND HYDROGENATED COTTONSEED OILS. Bauer, S. T.; and Gastinel, V., Jr. Oil & Soap 21(2): 36-42. 1944.

An investigation has been made of the relative effects of added metallic compounds normally found as contaminants in the tin bath upon the formation of free fatty acids and the rate of increase in the viscosity of heated palm and hydrogenated cottonseed oils. Salts of zinc and iron have been studied particularly. Rates of increase in viscosity of the two oils varied somewhat with the various salts. Results indicate that the inorganic salts are partly converted to ferric and zinc soaps in situ by reaction with free fatty acids already present or formed in the heated oils. The concentration at which the free fatty acids in the palm oil become constant is directly affected by the specific nature of the added metallic compounds. When hydrogenated cottonseed oil having a lower initial content of free fatty acids than the palm oil is treated in the same manner, there is an accumulation of free fatty acids during heating until a definite value is attained, after which it remains constant on further heating.

46. MODIFICATION OF VEGETABLE OILS. I. NEW PRODUCTS BY FRACTIONAL CRYSTALLIZATION OF GLYCERIDES FROM PETROLEUM NAPHTHA. Bailey, A. E.; Feuge, R. O.; Kraemer, E. A.; and Bauer, S. T. Oil & Soap 20(7): 129-132. 1943.

Solvent crystallization of cottonseed and peanut oils were explored as a means of producing winterized salad oils and an olive oil-like product from peanut oil to replace supplies greatly reduced by World War II. An apparatus that combined a chiller and filter was designed and

built to permit crystallization and crystal separation under strictly controlled conditions. Researchers succeeded in producing a modified peanut oil very similar to olive oil in most of its properties but quite superior in several important respects. The same equipment and techniques also produced a higher yield of a better quality solvent-winterized cottonseed oil than did the conventional solvent-winterization methods, in a fraction of the time required by the older method. These same techniques were successful in producing a solvent-winterized peanut oil, though it had not been possible to produce such an oil by the methods in use. Apparatus and techniques are described in detail, and analyses of the oils given.

37. LABORATORY DEODORIZER FOR FATS AND OILS. Bailey, A. E.; and Feuge, R. O. Ind. Eng. Chem. 15: 280-81. 1943.

A simplified apparatus for deodorization of fats and oils in the laboratory is described, with illustrative diagram.

36. HYDROGENATED COTTONSEED OIL AS A SUBSTITUTE FOR PALM OIL IN THE PRODUCTION OF TIN PLATE AND COLD REDUCED SHEET STEEL. Bauer, S. T.; and Markley, K. S. Oil & Soap 20(91): 1-11. 1943.

Palm oil is used to the extent of about 40 million pounds annually in the production of tin plate and terne plate. A domestic replacement for this product was desired, and research to develop such a product was initiated. Various types of selectively hydrogenated cottonseed oils were investigated, and one having an iodine number of approximately 50 seemed best adapted for use in the bath as a substitute for palm oil. The effect of heating on the palm oil substitute was determined. On the basis of these results and of evaluation of the products by producers of tin plate, specifications were formulated.

33. THE GRADING OF CRUDE VEGETABLE OILS BY MEANS OF REFINING TESTS--A REVIEW AND EVALUATION OF THE METHOD. Bailey, A. E.; Feuge, R. O.; and Bickford, W. G. Oil & Soap 19(6): 97-102. 1942.

For many years practically all of the crude cottonseed oil produced in the United States has been bought and sold on the basis of a laboratory alkali refining test. Methods and apparatus employed in this test are described. The test has been generally satisfactory, but changes in processing methods have affected its reliability and suitability for cottonseed oil, and difficulties have been encountered in applicability of the test for some other vegetable oils. Variations in oils from different producing sections and the probable introduction of other and more complex refining methods may also require new ways of grading. Further research on methods of grading oil for trading purposes is recommended.

31. VARIABLES AFFECTING THE YIELD OF NORMAL OLEIC ACID PRODUCED BY THE CATALYTIC HYDROGENATION OF COTTONSEED AND PEANUT OILS. Bailey, A. E.; Feuge, R. O.; and Smith, B. A. Oil & Soap 19(10): 169-76. 1942.

In order to hydrogenate cottonseed and peanut oils to obtain maximum production of normal oleic acid and minimum losses of potential oleic acid through formation of stearic or iso-oleic acids, it became necessary to investigate the effect of a number of processing variables on the reaction. Variables investigated include temperature, concentration of catalyst, pressure of the hydrogen, degree of agitation, and nature of the nickel catalyst. Formation of stearic acid is repressed and that of iso-oleic acid favored by increasing the temperature and catalyst concentration and decreasing pressure and degree of agitation. Nature of the nickel catalyst must have considerable effect on the composition of the hydrogenated product. In the hydrogenation of cottonseed oil, within a comparatively wide range of conditions, the production of total solid acids with a given catalyst is relatively constant. Peanut oil is more suitable than cottonseed oil as a raw material for production of normal oleic acid.

2836. PROCESS FOR PRODUCING SULFUROUS ACID-TREATED ALUMINA. Pons, W. A., Jr.; Kuck, J. C.; and Frampton, V. L. U. S. Pat. No. 3,158,578, November 24, 1964.

A bleaching process for the simultaneous removal of the Halphen test response, and lowering or reducing of the oil-soluble yellow or red and green chlorophyll-like pigments from refined cottonseed oil. This invention has as its object the production of bleached oils of light color which are negative to the Halphen test.

2826. PROCESS FOR PREPARING RANEY NICKEL CATALYST. Tumer, H. V.; and Cousins, E. R. U. S. Pat. No. 3,139,408, June 30, 1964.

This invention relates to a process for preparing an improved Raney nickel catalyst suitable for the hydrogenation of glyceride oils, one in which the hydrogenation is uniform and does not vary with the starting alloy. Another object of this invention is to provide a liquid medium for the storage of the prepared catalyst.

2369. PROCESS FOR BLEACHING OFF-COLOR COTTONSEED OILS. Pons, W. A., Jr.; and Kuck, J. C. U. S. Pat. No. 3,036,102, May 22, 1962.

A process for bleaching refined cottonseed oil to remove the fixed red color bodies from off-grade or unbleached cottonseed oils. The process involves treatment of the oils with an activated alumina of specified size, the activated alumina containing at least 10 percent by weight of moisture.

1484. COTTONSEED OIL COLOR REVERSION PREVENTION. Dechary, J. M.; Kupperman, R. P.; and Thurber, F. H. U. S. Pat. No. 2,787,625, April 2, 1957.

A process for preventing color reversion in crude cottonseed oils involving treatment of the crude oil with an amount of para-aminobenzoic acid sufficient to react with the gossypol and gossypol-like pigments contained in the oil. The insoluble reaction products can be removed mechanically.

1323. METHOD OF INHIBITING POLYMERIZATION AND PRODUCT. Bickford, W. G.; Mack, C. H.; and Moore, R. N. U. S. Pat. No. 2,729,671, January 2, 1956.

Norconidendrin and alpha- and beta-conidendrols are effective antioxidants and antipolymerization agents in preventing the oxidative deterioration, rancidification and polymerization in edible fats and oils, candies, marine oils, mineral oils, vinyl-type monomers, and synthetic rubber, and in products containing the above-mentioned materials.

1105. PROCESS FOR FRACTIONATING GLYCERIDIC MIXTURES OBTAINED AS HYDROCARBON SOLUTIONS. Skau, E. L. U. S. Pat. No. 2,684,378, July 20, 1954.

This is a method of winterizing a cottonseed oil by a solvent-fractional crystallization process that avoids the phase separation characteristic of cottonseed oil in moist solvents. The oil is dissolved in enough acetone and hexane to produce a solution of specified proportions, the solution is cooled to a specified temperature at which some of the higher melting glycerides solidify, and the solidified glycerides are centrifugally isolated from the solution.

1104. PROCESS FOR MODIFYING GLYCERIDIC FATS AND OILS. Skau, E. L. U. S. Pat. No. 2,684,377, July 20, 1954.

This is a method of winterizing a peanut oil by a solvent-fractional crystallization process that avoids the gelation characteristic of peanut oils. The oil is dissolved in enough acetone

and hexane to produce a solution of specified proportions, the solution is cooled to a specified temperature at which some of the higher melting glycerides solidify, and the solidified glycerides are centrifugally isolated from the solution.

1102. PROCESS FOR ALKALI-REFINING CRUDE NATURAL OILS. Markley, K. S.; and Feuge, R. O. U. S. Pat. No. 2,686,796, August 17, 1954.

This is a method of alkali-refining crude natural glyceride oils so that the color reduction is increased without increasing refining loss. A mixture of the oil and the amount and type of aqueous alkali usually employed is briefly exceedingly violently agitated. This agitation shears the liquids, and the mixture is processed in the usual manner.

OIL--NEW PRODUCTS

3292. A METHOD FOR ADSORBENT FRACTIONATION OF COTTONSEED OIL FOR EXPERIMENTAL INTRAVENOUS FAT EMULSIONS. Singleton, W. S.; Brown, M. L.; and Zeringue, H. J. J. Am. Oil Chemists' Soc. 43: 592-95. 1966.

Results of animal screening tests of emulsified cottonseed oils of the glanded and glandless varieties justify attributing some of the undesirable effects to pigments and minor polar components. A bleaching earth-alumina fractionation method was developed for removal of pigments and polar components of cottonseed oil whereby the triglyceride portion was obtained as an essentially pure, water-white fraction. Bleaching earth and chromatographic alumina used in sequence are very satisfactory adsorbents at concentrations of 20 weight percent of earth and a 1-to-1 weight ratio of alumina-to-oil. Thin-layer chromatography indicated the presence of sterol esters in the fractionated oil, but no polar components were detected.

3287. LAB-SIZED CRYSTALLIZER SEPARATES FAT IN MINUTES. Pominski, J.; Gallo, A. S.; and Spadaro, J. J. Food Eng. 38: 122-23. 1966.

The development of an economical continuous process for making cocoa butter-like fats from hydrogenated cottonseed oil requires a rapid method of separating desirable from undesirable fat fractions in the solvent miscella. For this purpose a small, vertical, batch-type rotary scraped-surface crystallizer was developed for bench-scale use. The unit occupies little space and requires as little as 1,700 ml. of solvent-fat miscella. Rates of crystallization for times as short as 40 seconds can be evaluated. It can also be used to study reactions and to mix and heat the materials.

3281. RAPID REPRODUCIBLE PROCEDURE FOR WAFERS OF DRIED FOODS, ESPECIALLY THOSE OF HIGH FATTY CONTENTS: A TOOL FOR COLORIMETRY. Berardi, L. C.; Martinez, W. H.; Boudreaux, G. J.; and Frampton, V. L. Food Technol. 20: 120-22. 1966.

A method is described for rapid reproducible manufacture of wafers of dried foods possessing uniform smooth surfaces. The method involves the distribution of the dry food sample between thin disks of Teflon in a die. After the die and its contents are evacuated for a short specified time, they are pressed quickly with a relatively low pressing load to form wafers that can be used in reflectance measurements. Wafers of the same food sample yielded nearly identical reflectance spectra. A high degree of reproducibility as measured by reflectance spectra was also obtained with wafers prepared by different operators with different presses. The method was found satisfactory for preparing wafers of dry foods of high fatty contents, such as lyophilized egg yolks and lyophilized peanut butter, as well as those of intermediate or low fatty content.

3046. N, N-DIALKYLAMIDES OF LONG-CHAIN FATTY ACIDS AS PLASTICIZERS. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 42: 941-44. 1965.

A number of N, N-dialkylamides have been prepared, characterized, and evaluated as plasticizers for poly(vinyl chloride-vinyl acetate) copolymer. Among these are the N-oleoyl derivatives of di-isopropyl, dibutyl, di-isobutyl, diamyl, dihexyl, diheptyl, dioctyl, di-2-ethylhexyl, and didecylamines. Also included are the N, N-dibutylamides of 2-ethylhexanoic, neodecanoic, neotridecanoic, palmitic, stearic, linoleic, erucic, ricinoleic, naphthenic, dimer, pinic, epoxystearic, animal, cottonseed, hydrogenated cottonseed, rapeseed, Limnanthes douglasii seed, and parsley seed acids. Optimum low-temperature plasticizing properties are achieved for the N-oleoyl derivatives of dibutyl, diamyl, and dihexyl amines. These low-temperature properties are comparable with those of the adipate and sebacate plasticizers without the adverse volatility characteristics of the adipates. Compatibility of the N, N-dialkyloleamides extends through the dihexyl derivative. A brief heat stability study of some selected plasticized polyvinyl chloride copolymer compositions indicates that the thermal stabilization of amide plasticizers is not an insurmountable problem.

2902. EFFECT OF SALINE ELECTROLYTE ON PARTICLE SIZE IN FAT EMULSIONS BY ELECTRONIC COUNTING. Singleton, W. S.; and Brown, M. L. J. Am. Oil Chemists' Soc. 42: 312-14. 1965.

The number of particles of specific diameters in the fat emulsions SR-151, Intralipid and Lipofundin, by electronic counting, was critically dependent upon the elapsed time of contact of the emulsions with the diluting saline electrolyte. The most pronounced change in the number of particles as a function of time occurred with the particles of smallest diameters, those less than 2μ. Increases in the number of particles by approximately 100 percent occurred within 20 minutes of mixing the emulsions and electrolyte. Apparently, coalescence or aggregation of smaller particles caused the increases in numbers. By extrapolation to zero time, accurate counts were obtained.

2901. SOLIDIFICATION OF COCOA BUTTER. Lovegren, N. V.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 42: 308-12. 1965.

When molded, cocoa butter does not behave like other confectionery fats. Explanations for the behavior of cocoa butter generally are unavailable. The linear contraction of molded cocoa butter on solidification under various conditions was determined. Maximum linear contraction of about 2 percent was measured when a well-seeded sample was solidified at 16° C. Nearly all of this contraction occurred during the first half hour. A theoretical explanation for this contraction was developed. Linear contraction takes place after the well-seeded cocoa butter has solidified in the next-to-highest melting form and while this solid form is transforming to the most stable polymorph. Additional information was developed on the polymorphic forms of cocoa butter, the permanence of seed crystals at various temperatures, rates of solidification, and solidification characteristics of unseeded cocoa butter. The data were obtained by examination of a small sample with a volumetric dilatometer and direct measurement of linear contraction during solidification in a mold.

2875. CHARACTERIZATION OF LIPIDS OF HUMAN SERUM FOLLOWING INTRAVENOUS FAT INFUSION. Benerito, R. R.; Singleton, W. S.; Calamari, L. D.; and White, J. L. Am. J. Clin. Nutr. 16: 173-81. 1965.

The object was to ascertain whether or not major changes in human serum proteins, lipids, or lipoproteins occurred during or following a single administration of an intravenous fat in the form of a stable emulsion. Serum obtained from blood samples withdrawn from each of five patients (1) immediately before starting on infusion, (2) at the midpoint of the infusion, (3) at the completion of the infusion, (4) 2 hours after completion of the infusion, and

(5) 4 hours after completion of the infusion were analyzed. Serum samples applied to wide paper strips were portioned electrophoretically and the electrophoreograms cut into three strips. One strip was used for determination of relative percentages and mobilities of protein fractions and another for determination of relative mobilities of lipids. Once the positions of lipids and proteins were ascertained, the third strip was used for elution of lipids from the electrophoreograms. Types of lipids in each of the five sections were characterized chromatographically. Total lipids extracted from whole sera before electrophoresis were determined quantitatively and characterized chromatographically. Total nitrogen, nonprotein nitrogen, and albumin/globulin ratios of all sera samples were also determined. Although small changes in various protein and lipid fractions were noted during the course of a single infusion, no lasting effects were observed.

2874. CHARACTERIZED OILS FOR USE IN INTRAVENOUS FAT EMULSIONS. Singleton, W. S. Am. J. Clin. Nutr. 16: 16-22. 1965.

Several natural oils were obtained and processed in the laboratory by various procedures. These oils included a cold-pressed peanut oil extracted from peanuts of excellent quality, a prepressed peanut oil obtained commercially, a prepressed solvent-extracted peanut oil, cottonseed oil, soybean oil, and various rice, sesame, and olive oils. In addition, synthetic glycerides of widely varying compositions were prepared, including "simulated" human fat, and mixed acetoglycerides. All of these products were made available for testing in emulsions for intravenous nutrition. The surface tension and interfacial tension against water of the natural and synthetic oils were determined. Interfacial tensions of the various glycerides do not differ greatly, and none are low enough for spontaneous emulsification. Added emulsifiers are required. On the basis of available results, peanut oil gave the most unsatisfactory physiological results. The synthetic fats tested were utilized metabolically and showed no significant differences from the better natural oils in the incidence of adverse clinical effects. Cottonseed oil selected for resistance to oxidative changes was equal or superior to all of the other oils tested, natural or synthetic.

2846. CHROMATOGRAPHICALLY HOMOGENEOUS LECITHIN FROM EGG PHOSPHOLIPIDS. Singleton, W. S.; Gray, M. S.; Brown, M. L.; and White, J. L. J. Am. Oil Chemists' Soc. 42: 53-56. 1965.

Chromatographically homogeneous egg lecithin, as determined by TLC on Silica Gel G, has been isolated from crude egg phosphatides by column chromatography on alumina through modification of existing methods. The modified method involves application of crude egg phosphatides to a column of alumina in the proportion of 1 g. phosphatide 25 g. alumina, and elution of the lecithin fraction with the two-component solvent system, chloroform:methanol, 9:1 by volume. This method of purification separated lecithin from other choline and non-choline components of crude phosphatides, avoided overloading of the alumina column, and made unnecessary the need for a second chromatographic fractionation on silicic acid, which is needed in existing methods for purification of lecithin. The use of fresh yolks permitted easier removal of pigment from the final product than was possible with commercially dried yolks. Phosphatides extracted from dried yolks were much more highly colored than were the phosphatides extracted from fresh yolks and the color persisted through chromatography on alumina. The fatty acid/phosphorus (FA/P) molar ratio of the purified lecithin was 2.00, which is the theoretical FA/P molar ratio of phosphatidylcholine; other materials with this ratio were not present.

2818. THE PREPARATION AND PLASTICIZING CHARACTERISTICS OF THE N, N-bis(2-ALKOXYETHYL) AMIDES OF LONG-CHAIN FATTY ACIDS. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 41: 781-82. 1964.

A number of N-acyl derivatives of bis(2-alkoxyethyl) amines have been prepared, characterized, and screened as plasticizers for poly(vinyl chloride-vinyl acetate) copolymer. Included were the

N,N-bis(2-alkoxyethyl)amides of saturated (normal and branched), unsaturated, epoxy, dimer, terpenic, and naphthenic acids, as well as mixed acids from animal fats, rapeseed, Limnanthes douglasii seed, parsley seed, and selectively hydrogenated cottonseed oils. With the exception of the linoleic acid derivative, all these amides, including the stearamide derivative, were compatible. Some exhibited low-temperature properties comparable with those of the adipate plasticizers without the adverse volatility characteristics of the adipates. With the exception of the epoxystearamide derivative, the N,N-bis(2-alkoxyethyl)amides were inferior to dioctyl phthalate in thermal stability. The N,N-bis(2-ethoxyethyl)stearamide is of special significance in that it seems to be the only carboxylic derivative of stearic acid known to be effective as a primary plasticizer.

2803. PREPARATION OF PARTIAL GLYCERIDES BY DIRECT ESTERIFICATION. Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 41: 727-31. 1964.

A previously described procedure for the direct esterification of diglycerides without interesterification occurring has been evaluated for the preparation of mono- and diglycerides. The esterifications were accomplished with p-toluenesulfonic acid catalyst and with continuous removal of water of esterification by azeotropic distillation. The effect of such variables as unsaturation, chain length, mode of addition of solvent, and reaction temperature on the composition and yield of glycerides was observed. Chemical and chromatographic analyses were used to determine the composition of the glycerides and their component fatty acids and to detect the presence of isomeric glycerides. Simple esterification of 1-monostearin with oleic acid at 80° C. yielded as much as 72.3 percent diglycerides, and esterification of glycerol with stearic acid at 100° C. yielded up to 70.1 percent monoglycerides, each calculated on a glyceride basis. It is concluded that simple esterification predominates with some intra- and interesterification occurring.

2790. CHROMATOGRAPHICALLY HOMOGENEOUS EGG LICITHIN AS STABILIZER OF EMULSIONS FOR INTRAVENOUS NUTRITION. Zeringue, H. J.; Brown, M. L.; and Singleton, W. S. J. Am. Oil Chemists' Soc. 41: 688-91. 1964.

Physically stable emulsions of cottonseed and soybean oils at 20 percent concentration were prepared with chromatographically homogeneous egg lecithin at 1 percent concentration as the sole emulsifier and a 2.5 percent solution of glycerol as the aqueous phase. The physical stability of the emulsions was a function of the pH of the product; optimum pH was 6.6 to 6.8. Aqueous solutions of dextrose and sorbitol decreased in pH to 4.8 when autoclaved, regardless of prior adjustment of pH to as high as 8.5, and emulsions in which these solutions were used as the aqueous phase exhibited phase separation. There was no significant decrease in pH and no phase separation in emulsions that contained glycerol solution as the aqueous phase. It appears that glycerol is superior to dextrose as the isotonic agent in lecithin-stabilized emulsions for intravenous nutrition. Particle sizes and their distribution in lecithin-stabilized emulsions of cottonseed and soybean oils were determined by means of a Coulter Counter. Approximately 99 percent of the oil in these emulsions was dispersed as particles whose diameters were no larger than 5μ.

2767. NEW PROCESS OFFERS ECONOMICAL CANDY FATS. Feuge, R. O.; Gajee, B. B.; and Lovegren, N. V. Candy Ind. & Confectioners J. 122(13): 7, 8, 10, 14. 1964.

The preparation of cocoa butter-like fats by the direct combination of oleic acid and diglycerides of palmitic and stearic acids was investigated. Operating conditions were found that should be suitable for use in large-scale preparations. The reaction products consisted of about 80 percent of the components desired in good cocoa butter-like fats. The undesirable components, trisaturated glycerides, were removed by fractional crystallization from a solvent. These cocoa butter-like fats have properties desired in a confectionery fat, including a short melting range, brittleness at room temperature, and good compatibility with cocoa butter.

2709. CONFECTIONERY FATS: THEIR CURRENT STATUS AND POTENTIAL MARKET. Feuge, R. O. J. Am. Oil Chemists' Soc. 41: 4, 26, 30, 63. 1964.

One can predict that the production and use of confectionery fats will expand. A potential market of 100 million lb. per year is not overly optimistic, especially considering the fact that potential utilization in so-called pastel coatings, which have not been discussed, are not included in this amount. The preferred physical characteristics of confectionery fats have been well established, and in their most important respects are the characteristics of cocoa butter. Fats made from lauric acid oils can be prepared readily and have a number of the preferred characteristics; therefore, their use will continue for many years. Fats made from oleic-palmitic-stearic acid oils have been produced up to now in relatively small quantities because of more involved processes and resulting higher cost, but eventually the utilization of large quantities of very good products of this type can be expected. The future role of fatty products that resemble confectionery fats in physical properties but differ in not being glycerides is uncertain but probably will not be great.

2693. THE PREPARATION AND PLASTICIZING CHARACTERISTICS OF PIPERIDIDES OF LONG-CHAIN FATTY ACIDS AND N-FATTY ACYL DERIVATIVES OF OTHER CYCLIC IMINES. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 41: 237-40. 1964.

Forty-six N-acyl derivatives of cyclic imines have been prepared, characterized, and screened as primary plasticizers for poly(vinyl chloride-vinyl acetate) copolymer. Among these were the piperidides of decanoic, palmitic, stearic, oleic, erucic, ricinoleic, epoxystearic, epoxyoleic, diepoxystearic, sebacic, pinic, cottonseed, hydrogenated cottonseed, rapeseed, Limnanthes douglasii, animal, 2-ethylhexanoic, naphthenic, and dimer acids, as well as the N-oleoyl derivatives of a number of substituted piperidines and other cyclic imines including pyrrolidine, piperazine, hexamethylenimine, tetrahydroquinoline, 3-azabicyclo [3.2.2] nonane, dipyridylamine, and carbazole. In general these amides of the 5-, 6-, and 7-membered cyclic imines exhibited exceptionally high plasticizing efficiencies. The compatibilities observed were the best to date for fatty acid derivatives on the basis of both individual and ternary fatty acid composition-compatibility data. Several of these amides exhibited low-temperature characteristics in the adipate plasticizer range without the adverse volatility characteristics of the adipates. There are indications that some of them have appreciable anti-fungal activity. It has been concluded that cyclic imines will, in general, produce fatty acid derivatives of better than average compatibility as vinyl plasticizers.

2647. A NUTRITIONAL EVALUATION OF ACETOSTEARINS IN RATS. Coleman, R. D.; Gayle, L. A.; and Alfin-Slater, R. B. Contractor: Department of Biochemistry and Nutrition, University of Southern California, Los Angeles, Calif. J. Am. Oil Chemists' Soc. 40: 737-42. 1963.

A modified glyceride, acetostearin, has been studied in feeding experiments with rats. As acetostearins are more readily absorbed than the standard fats from which they are prepared, it was possible also to study the effect of high levels of saturated fat on utilization of essential fatty acid. The retarded growth and poor survival of acetostearin-fed animals indicate the possibility of antagonism of the acetostearins to essential fatty acid utilization. The decreased plasma cholesterol levels and elevated liver cholesterol values observed in the acetostearin-fed animals appear to be due mainly to essential fatty acid deficiency. High cholesterol levels in the adrenals show some indication of the involvement of caloric insufficiency in cholesterol metabolism of acetostearin-fed rats. Therefore, the probable explanation of the poor nutritional performance of rats fed acetostearin is a combination of essential fatty acid deficiency and caloric insufficiency.

2623. IN MECHANICAL TEMPERING LIQUID FAT HAS TO BE IN SUPERCOOLED STATE. Feuge, R. O.; Bradshaw, B. L.; and Lovegren, N. V. Candy Ind. & Confectioners J. 120(13): 7-8, 10-12. 1963.

Further work was conducted on a new procedure formaking cocoa butter-like fat. This procedure consists of directly combining partial glycerides and fatty acids (esterification) and is potentially capable of a 100 percent yield of product; that is, all of the fat formed should resemble cocoa butter. In practice, the water generated when partial glycerides and fatty acids are combined acts as a rearrangement catalyst, and some rearrangement of the cocoa butter-like fat occurs. However, some relatively good products have been prepared in the laboratory. Different modifications of the new procedure for producing cocoa butter-like fats investigated during the current year are described. The previously described process for making cocoa butter-like fat by the random rearrangement of completely hydrogenated cottonseed oil and a triolein product, followed by fractionation, was further investigated and yielded a relatively good but expensive fat. The results indicate that cost of the fat can be reduced. An analytical procedure for determining quantitatively the contents of trisaturated and monounsaturated, diunsaturated, and triunsaturated triglycerides in fats was evaluated and modified to adapt it to the analysis of cocoa butter and similar fats. The process of tempering cocoa butter and chocolate by intense mechanical working was further investigated. It was discovered that liquid fat cooled quickly to below its highest melting point (supercooled liquid) could be converted readily to the highest melting crystal form (tempered) by intense mechanical working. The presence of liquid crystals in the melted fat is believed to make this possible. The linear contraction of cocoa butter on molding and solidification was investigated. Seeded cocoa butter did not contract when solidified. Contraction commenced only when the bulk of the solidified fat started to change from the second highest to the highest melting form. Contraction ceased when the conversion was halfway complete. Maximum linear contraction was about 2 percent.

2616. CALORIC AVAILABILITY AND DIGESTIBILITY OF NEW-TYPE FATS. Booth, A. N.; and Gros, A. T. J. Am. Oil Chemists' Soc. 40: 551-53. 1963.

Digestibility and caloric availability of new-type fats were determined by feeding these products to young rats on a restricted caloric intake. Amylose stearate, amylose palmitate, amylose oleate, distearin adipate, and glycerol adipate were low in both categories. Diolein fumerate was completely digested but poorly utilized. These materials were compared with common oils and fatty acids and have potential use as pan greases and surface coatings for foods.

2615. N, N-BIS(2-ACYLOXYETHYL) AMIDES OF LONG-CHAIN FATTY ACIDS AS PLASTICIZERS. Magne, F. C.; Mod, R. R.; and Skau, E. L. J. Am. Oil Chemists' Soc. 40: 541-45. 1963.

A series of N, N-bis(2-acyloxyethyl) amides and a few related derivatives has been prepared, in which a long-chain fatty acid or fatty acid mixture comprises 33 mole percent of the total acyl groups present; the other 66 mole percent being either a short-chain aliphatic or aromatic acyl group. Both ordered and random diesteramides have been prepared. All have been tested as plasticizers for vinyl chloride copolymers. Some are acceptable as nitrile rubber softeners and cellulose acetate plasticizers. The ordered diesteramides give the best overall performance as plasticizers, but it is possible to achieve acceptable performance levels in semiordered diesteramides.

2567. PREPARATION OF GLYCERIDES BY CONTROLLED ESTERIFICATION. Feuge, R. O.; Willich, R. K.; and Guice, W. A. J. Am. Oil Chemists' Soc. 40: 260-64. 1963.

A simple procedure for esterifying glycerides without interesterification would be highly useful for preparing on a large scale a number of tailor-made fats, including cocoa butter-like

fats. Such esterifications were carried out by employing ρ-toluenesulfonic acid as catalyst and continuously removing the water of esterification by azeotropic distillation with aliphatic hydrocarbons or by stripping with vaporized hydrocarbons. Even though ρ-toluenesulfonic acid rapidly disproportionated 1-monostearin, even at 120° C., apparently esterification was faster and only a moderate amount of ester-ester interchange and acidolysis occurred. Diacid glycerides might be prepared from monoglycerides by the procedure used. Saturated diglycerides were esterified with oleic acid with little ester-ester interchange or acidolysis occurring; however, intra-esterification was extensive. The reaction between 1,3-distearin and oleic acid yielded approximately 75 percent 1-oleodistearin and 25 percent 2-oleodistearin. Saturated diglycerides were esterified with sebacic acid, again with little or no interestification.

2528. SUSPENSIONS OF HIGH-MELTING TRIGLYCERIDES. White, J. L.; and Singleton, W. S. J. Am. Oil Chemists' Soc. 40: 186-88. 1963.

A method for preparing stable oil/water suspensions of cottonseed stearine, tristearin, tripalmitin, trimyristin, methyl stearate, and palmitic acid in concentrations up to 10 percent with minimum concentrations of stabilizing agents is described. Using 2.5 percent of polyethylene glycol 400 monostearate (based on weight of hard fat), 0.1 percent of Pluronic F 68, and 0.2 to 0.25 percent of Carbopol 934 (the concentrations of these two latter agents are based on the weight of the aqueous phase), we prepared suspensions of the hard fats by simple stirring. They were stable for at least one month at room temperature and could be sterilized. The size of the dispersed fat particles was 20 to 40μ. Apparent viscosities of cottonseed stearine suspensions at 2-, 5-, and 10-percent concentrations were 3.59, 5.95, and 6.62 poises, at 25° C., respectively. Suspensions as described should be useful in those areas of investigation in which solid fatty materials in the form of stable dispersions are desirable.

2498. NEW INTRAVENOUS FAT EMULSION. Cohn, Isidore, Jr.; Singleton, W. S.; Hartwig, Q. L.; and Atik, M. J. Am. Med. Assoc. 183: 755-57. 1963.

A new intravenous fat emulsion has been developed from cottonseed oil and synthetic emulsifiers. The emulsion can be infused into dogs over an extended period with minimal side effects, even at dosages four to five times that recommended clinically. Eleven of 14 dogs tolerated 30 infusions of the improved SR 695 emulsion at a dosage of 40 ml. per kg. per 24 hours (6 gm. cottonseed oil per kg. body weight). The dogs generally gained weight during the infusion period. Laboratory data remained within normal limits except for transient rise in white blood cell count and slight drop in hematocrit. The intravenous fat pigment was found in liver biopsies taken after termination of the infusion program. There appears to be less pigment formation than with use of previous intravenous fat emulsions. Preliminary clinical testing of the emulsion is underway. Possible limitations in the use of intravenous fat emulsions without adequate amino acid or carbohydrate supplementation are discussed.

2442. DERIVATIVES OF FATS FOR USE AS FOODS. Feuge, R. O. J. Am. Oil Chemists' Soc. 39: 521-27. 1962.

Suitable methods of manufacture always have been major factors in the development of new edible products from fats. A review of the chemistry involved in the preparation of various surface-active agents, including monoglycerides, lactoglycerides, polyglycerol esters, and sugar esters, reveals a number of problems in controlling the course of the reaction to obtain economically products of reasonable purity and uniformity. Suggested innovations in the manufacture of some of these products are described. The production of specialty fats also presents some problems in chemistry. Because of such problems, cocoa butter-like fats have heretofore not been made from domestic oils. A recent discovery that may change this situation is discussed. The manufacture of specialty fats such as the acetoglycerides and

brominated oils is described. A number of new derivatives of fats made in the laboratory possess useful properties but are not being manufactured. These are discussed from the standpoint of the chemistry involved in their preparation.

2393. PROGRESS ON THE DEVELOPMENT OF CONFECTIONERY FATS. PART II. A NEW PROCEDURE FOR MAKING COCOA BUTTER-LIKE FATS. (Published: cocoa butter-like fat. . . New Esterification Procedure shows Promise.) Feuge, R. O.; Landmann, Werner; and Lovegren, N. V. Candy Ind. Technol. 119(3): 15-16. 1962.

A new approach to the preparation of cocoa butter-like fats involves a procedure for esterification under conditions where all traces of moisture formed during the esterification reaction are removed as soon as they are formed and the random rearrangement of the fatty acids is prevented. 1,3-Distearin esterified with pure oleic acid yielded over 90 percent oleodistearin, of which about three-fourths was 1-oleodistearin. Several cocoa butter-like fat products were prepared by esterifying oleic acid with the diglycerides of palmitic and stearic acids and fractionating the product by crystallization from hydrocarbon solvent. Physical properties agreed closely with those of cocoa butter. The newly discovered esterification procedure should be good for making cocoa butter-like fats from diglycerides which can be manufactured readily on a large scale. Additional work is underway to establish the best conditions for operating the process.

2392. PROGRESS ON THE DEVELOPMENT OF CONFECTIONERY FATS. PART I. TEMPERING OF CONFECTIONERY FATS AND COATING COMPOSITIONS. (Published: Conf. fat research shows. . . Mechanical Method May Give Tempered Effect.) Feuge, R. O.; Landmann, Werner; and Lovegren, N. V. Candy Ind. Technol. 119(2): 7-9. 1962.

Fats in confectionery are tempered to develop the most desirable size and type of fat crystals so that the confectionery coating will be firm, glossy, of a fine surface texture, and resistant to fat bloom. Tempering usually consists of holding the coating for a short time at temperatures just below the melting point of the fat, followed by cooling. It has been observed that crystal modifications similar to those produced by tempering will occur on mechanical working of quickly solidified crystals of fat. The mechanical working could be accomplished by forcing the fat repeatedly through small orifices at pressures up to 1,000 p. s. i. It is believed that through use of this discovery improvements in the manufacture of chocolate and chocolate-type confections can be accomplished. Probably the use of tempering kettles can be eliminated, the enrobing machines can be simplified, and a truly continuous process can be devised in which the material in actual process is perfectly tempered and always small in amount. In such a modified process crushed or granulated chocolate would be fed continuously into a heat exchanger where the chocolate would first be melted and then cooled until some unstable fat crystals formed. This mixture would then be forced through a series of orifices and pumped to the enrober or molding machine.

2377. TEMPERING TRIGLYCERIDES BY MECHANICAL WORKING. Feuge, R. O.; Landmann, Werner; Mitcham, D.; and Lovegren, N. V. J. Am. Oil Chemists' Soc. 39: 310-13. 1962.

The tempering of fat products to convert their components to stable polymorphs is an important and a sometimes troublesome problem in the manufacture of these products, particularly chocolate and chocolate-type confections. A solid-to-solid transformation to the stable polymorphs can be effected by mechanical working consisting of extrusion under pressure. With a fat of relatively few components, such as cocoa butter, evidence of the transformation can be obtained from X-ray diffraction patterns. For more complex fats, hardness and melting characteristics must be considered. There is evidence that mechanical working is also effective in the transformation of cocoa butter-like fat made from hydrogenated cottonseed oil and

olive oil and in the transformation of highly hydrogenated cottonseed oil. Mechanical working to effect polymorphic transformation is also effective with products containing the fats mentioned.

2328. AN EMULSIFIER SYSTEM FOR EXPERIMENTAL INTRAVENOUS FAT EMULSIONS. Singleton, W. S.; White, J. L.; DiTrapani, L. L; and Brown, M. L. J. Am. Oil Chemists' Soc. 39: 260-63. 1962.

An emulsifying system has been developed for a fat emulsion that has been very satisfactory for intravenous administration to dogs. The emulsion contained cottonseed oil (15 percent), polyethylene glycol monopalmitate (1. 2 percent), tartaric acid ester of monoglycerides (0. 3 percent), and polyoxyethylene-polyoxypropylene (0. 3 percent) in isotonic dextrose solution. The polyethylene glycol monopalitate was isolated from laboratory and commercial esterification reaction mixtures of monoester, diester, and nonreacted polyethylene glycol. Washing with NaCl solution removed nonreacted glycol, and fractionation in acetone separated the mono- and diesters. The diester separated as a solid. Analytical methods were applied to characterize all fractions and to determine the reproducibility of composition of successive batches. The monopalmitates from laboratory and commercial preparations were similar in properties. The tartaric ester of monoglycerides was washed with NaCl solution and fractionated in acetone. The liquid portion was recovered and used for emulsification. Emulsions prepared with the fractionated emulsifiers appear to give satisfactory physiologic results when infused in dogs.

2300. COCOA BUTTER-LIKE FAT FROM COTTONSEED OIL: PRELIMINARY COST STUDY. Decossas, K. M.; Kolton, S. P.; Spadaro, J. J.; Feuge, R. O.; Pollard, E. F.; and Patton, E. L. J. Am. Oil Chemists' Soc. 39: 146-50. 1962.

This paper presents a preliminary cost study for producing cocoa butter-like fat from hydrogenated cottonseed oil and triolein at an annual rate of 8. 4 million lb. in a hypothetical plant, employing interesterification and fractional crystallization as used in pilot-plant studies at the Southern Utilization Research and Development Division. Equipment costs, total plant cost, manufacturing costs, and general expenses are given. Operating cost is estimated at 36. 9¢lb. of product. This included 31. 8¢ for manufacturing, 4. 1¢ for general expenses, and 1¢ for refining of the recycled mixture of saturates and unsaturates.

2275. PROPERTIES OF THE FATTY ACID ESTERS OF AMYLOSE. Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 39: 19-24. 1962.

To help establish the suitability of esters of amylose and fatty acids as dip-coating materials, esters of the even-numbered, saturated fatty acids, acetic through stearic, and of oleic and cottonseed oil fatty acids were prepared and their properties determined. The various esters generally were prepared by the interaction of acid chlorides, with the amylose suspended in a suitable mixture of solvents. Analyses for content of free fatty acid groups indicated that high proportions of the hydroxyl groups were esterified. Intrinsic viscosity decreased as the chain length of the acyl group increased, a reflection of the changing nature of the products rather than a decrease in the degree of polymerization of the amylose. As the chain length of the saturated fatty acid group increased, the softening point at first dropped and then rose, the minimum being obtained with the myristate. The stearate softened at 56. 2° C.; it had a hardness index of 139, about equal to the index of 140 for completely hydrogenated cottonseed oil. Densities were greater than those of the corresponding triglycerides and decreased as the chain length of the fatty acid group increased. Permeability to water vapor also decreased as the chain length increased, the index being 620×10^{-12} for the acetate and 15×10^{-12} for the stearate. The acetate had the highest tensile strength, about 4. 40 kg/sq. /mm., whereas the palmitate had a tensile strength of about 0. 49 kg. /sq. /mm. Elongation at the break point varied widely, being 632 percent for the caprate and 0. 95 percent for the stearate.

2247. DILATOMETRIC PROPERTIES OF SOME GLYCERIDES OF CONFECTIONERY FATS. Landmann, Werner; Lovegren, N. V.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 38: 681-85. 1961.

Two triglycerides, 1-oleodistearin, and 2-palmito-oleostearin, which are components of some confectionery fats, were synthesized and their melting behavior and dilatometric properties were determined. Expansion and melting dilations of the various polymorphic forms were measured. 1-Oleodistearin was found to have two melting points, 30.8 and 42.1° C., while 2-palmito-oleostearin was found to have melting points at 24, 37, and 40.5°. The rate of transformation of the thermodynamically unstable polymorphs at temperatures below their melting points was much more rapid than those for the corresponding 2-oleo isomers previously reported. Mixtures of 2-oleopalmitostearin with 2-palmito-oleostearin and 1-oleodistearin with 2-oleodistearin were examined dilatometrically. In these mixtures the components apparently do not temper at the same rate to similar polymorphic forms and thus there is some degree of incompatibility even though in each of these mixtures the components are positional isomers. The properties of the intermediate melting mixtures depend on the method of tempering.

2241. INTERACTIONS IN FAT EMULSION-PROTEIN SYSTEMS. Mann, G. E.; Brown, M. L.; DiTrapani, L. L.; and Singleton, W. S. Metabolism, Clin. and Exptl. 10: 859-68. 1961.

Two cottonseed oil emulsions of the type proposed for intravenous alimentation were incubated with bovine serum albumin (BSA) solutions and with dog blood serum. One emulsion (stabilized in part with soya phosphatides) was spontaneously creamed by BSA and bound considerable amounts of BSA nitrogen, whereas the second emulsion (stabilized by synthetic nonphosphatide emulsifiers) was not creamed by BSA and bound only insignificant amounts of BSA nitrogen. Neither emulsion was spontaneously creamed by dog blood serum. The phosphatide-stabilized emulsion bound relatively larger quantities of nitrogen from dog serum than did the nonphosphatide-stabilized emulsion and also caused more disturbance in the serum protein fraction than did the nonphosphatide emulsion. Possibly the greater tolerance of dogs for the nonphosphatide emulsion is related to the fact that it disturbs blood serum protein relationships less than the phosphatide emulsion.

2199. CONFECTIONERY FATS. II. CHARACTERIZATION OF PRODUCTS PREPARED BY INTERESTERIFICATION AND FRACTIONATION. Landmann, Werner; Lovegren, N. V.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 38: 466-69. 1961.

Several cocoa butter-like fats were prepared by fractional crystallization of the reaction product obtained by interesterifying highly hydrogenated cottonseed oil and a triolein product or olive oil. These fats were characterized and compared with cocoa butter. The fats obtained by fractional crystallization from acetone solutions contained varying amounts of glycerides melting above 37° C. This is an undesirable feature which caused the fats to thicken too much when used in chocolate-type compositions under the same conditions employed with cocoa butter. The higher-melting glycerides could be removed by filtration, or their proportions could be decreased by changing the fractionation temperatures. Most of the fats melted over the same temperature range associated with cocoa butter, and the best of them resembled cocoa butter closely over the temperature range, 0° to 30° C. and in hardness at all test temperatures. The fats were reasonably compatible with cocoa butter; that is, in mixtures of the two, one did not cause extensive premelting of the other. According to their cooling curves, the cocoa butter-like fats did not supercool as cocoa butter does. The former contain not only the 2-oleodisaturated glycerides of cocoa butter, but also positional isomers of these glycerides. When the fats were molded under the same conditions employed with cocoa butter, linear shrinkage was only about one-third that of cocoa butter.

2198. CONFECTIONERY FATS. I. PREPARATION BY INTERESTERIFICATION AND FRACTIONATION ON PILOT-PLANT SCALE. Spadaro, J. J.; Lovegren, N. V.; Feuge, R. O.; and Patton, E. L. J. Am. Oil Chemists' Soc. 38: 461-65. 1961.

A cocoa butter-like fat has been prepared on a pilot-plant scale by the interesterification of hydrogenated cottonseed oil and a triolein product or olive oil, followed by fractional crystallization from acetone at two different temperatures. The coproducts--a fraction that consists primarily of trisaturates and is obtained by fractionation at 20 to 28° C. and another fraction that consists primarily of di- and triunsaturates and is obtained from the low temperature (0° C.) filtrate--are reused in the process. In five of the six pilot-plant runs conducted, 100 pounds of 70:30 or 75:25 mixtures of the hard fat and liquid oil were used as starting materials. In the sixth run, 140 pounds were used. Yields varied from 25 to 35 percent. Characteristics of the cocoa butter-like fat products are discussed. Variations in the products were made by changing the ratio of starting materials and by lowering the first crystallization temperature. Operational data obtained show that the process is commercially feasible. Although time to attain crystallization temperature was about 4 hours in the pilot-plant operations, laboratory data indicate that comparable products can be obtained through crystallization times as low as one-half hour. The steps in the process are considered conventional in commercial processing.

2176. COCOA BUTTER-LIKE SAMPLE. I. COATING WITH FAT NO. 5 HAS BETTER TEMPER. COCOA BUTTER-LIKE SAMPLE. II. FAT NO 5 SHOWS LOW-BLOOM TENDENCIES. Feuge, R. O.; Landmann, Werner; and Lovegren, N. V. Candy Ind. & Confectioners J. 117(1): 34-35; (2): 7-8. 1961.

Progress in the development of a cocoa butter-like fat from domestic oils is described. An improved cocoa butter-like fat (No. 5) was prepared by modifying the procedure of interesterifying completely hydrogenated cottonseed oil with olive oil and separating the oleodisaturated glycerides by fractional crystallization. The separated fat melted mostly over the temperature range for cocoa butter, but contained less liquid phase below 68° F. and about 3.7 percent solids above 95° F., which is the approximate melting point of cocoa butter. Other physical characteristics were determined. It was concluded that a reasonably good cocoa butter-like fat can be prepared from domestic oils by an economically feasible process. Although the cocoa butter-like fat was not an exact duplicate of cocoa butter and in some respects was less satisfactory, it was more desirable in some other respects. Among the more important variations was relatively small difference in performance when conditions of melting and solidifying were changed.

2133. FATTY ACID MORPHOLIDES AS PLASTICIZERS FOR VINYL CHLORIDE RESINS. III. TERNARY COMPATIBILITY-COMPOSITION DIAGRAMS FOR THE OLEIC-LINOLEIC STEARIC AND OLEIC-LINOLEIC-PALMITIC SYSTEMS. Magne, F. C.; Mod, R. R.; and Skau, E. L. J. Am. Oil Chemists' Soc. 38: 294-96. 1961.

A systematic investigation has been made of the effect of fatty acid composition on the compatibility of the corresponding mixed morpholides as plasticizers for vinyl chloride-vinyl acetate copolymer resins. Plasticizer evaluation data were obtained for the morpholides of lauric, palmitic, elaidic, and epoxyoleic acids and compared with the literature values for other fatty acid morpholides and mixtures. Ternary compatibility-composition diagrams have been constructed for the oleic-linoleic-stearic and the oleic-linoleic-palmitic morpholide systems. These diagrams make it possible to predict what adjustment, if any, must be made in the composition of any mixture of these fatty acids so that the corresponding morpholide mixture will be compatible. The diagrams will be useful in selecting the most appropriate and economical fatty acid sources from which to make compatible morpholide mixtures.

2132. FATTY ACID MORPHOLIDES AS PLASTICIZERS FOR VINYL CHLORIDE RESINS. II. THE MORPHOLIDES OF SELECTIVELY HYDROGENATED AND OF EPOXIDIZED COTTONSEED FATTY ACIDS. Magne, F. C.; Mod, R. R.; and Skau, E. L. J. Am. Oil Chemists' Soc. 38: 291-93. 1961.

The morpholides of selectively hydrogenated cottonseed acids and of epoxidized cottonseed acids have been prepared and shown to be good primary plasticizers for vinyl chloride homopolymer and copolymer resins. They are also acceptable plasticizers for cellulose triacetate and possibly also for Buna N softeners. The degree of hydrogenation or partial epoxidation was approximately equivalent to the conversion of all the polyunsaturated acyls to monounsaturated acyls.

2077. METABOLIC STUDIES OF GLYCERIDE ESTERS OF ADIPIC ACID. Shull, R. L.; Gayle, L. A.; Coleman, R. D.; Alfin-Slater, R. B.; Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 38: 84-86. 1961.

Data on the digestibility, absorption, and in vivo oxidation of two types of adipic acid esters of glycerides, a diglyceride adipate and a polymer of fatty acids, adipic acid, and glycerol, have been presented. Findings indicate that both of these compounds have high digestibility coefficients and that the stearic acid moiety is well absorbed. However, although the stearic acid moieties are oxidized slowly in both cases, which is compatible with previous findings that a slow rate of in vivo oxidation of the stearic acid moiety of glycerides obtains, the rate of oxidation of the stearic acid is greater when fed as the diglyceride adipate than as the polyester.

2046. MELTING AND DILATOMETRIC BEHAVIOR OF 2-OLEOPALMITOSTEARIN AND 2-OLEODISTEARIN. Landmann, Werner; Feuge, R. O.; and Lovegren, N. V. J. Am. Oil Chemists' Soc. 37: 638-43. 1960.

The glycerides, 2-oleopalmitostearin and 2-oleodistearin, which are important components of confectionery fats, were synthesized, and their melting behavior and dilatometric properties were determined. Each glyceride was found to have four melting points: 18.2, 24.5, 33.0, and 37.4° C. for 2-oleopalmitostearin; 22.8, 30.0, 37.7, and 42.8° C. for 2-oleodistearin. At temperatures just below the melting point, the rate of transformation of the thermodynamically unstable polymorphs varied from a few seconds to more than 8 days. For each glyceride, expansivities were determined for three polymorphs and melting dilations for two polymorphs. Mixtures of the two glycerides were examined dilatometrically and found to behave in some respects as single components. The mixtures were readily tempered so that both components were in the same polymorphic form, and expansivities and melting dilations were obtained for mixtures in three polymorphic forms. Dilatometric data also were obtained for a sample of cocoa butter and a sample of sweet milk chocolate of the coating type.

2037. PRODUCTION OF SPECIALTY EDIBLE FATS. Feuge, R. O. J. Am. Oil Chemists' Soc. 37: 527-32. 1960.

This paper takes up several more or less arbitrarily selected products and processes. The requirements of cocoa butter-like fats are discussed in relation to the properties and composition of cocoa butter. The suitability of cocoa butter-like fats from lauric acid oils and from oleic and linoleic acid oils is compared. The need for and preparation of oils having exceptional resistance to deterioration is discussed, with emphasis on a laboratory preparation consisting of a mixture of dibutyropalmitins and dibutyrostearins. Several potentially useful fats and oils are mentioned, including dibasic acid-containing fats, products from polyols other than glycerol, and acetoglycerides.

2035. METHYL ESTERS DIRECTLY FROM ACIDULATED COTTONSEED SOAPSTOCK. PRELIMINARY COST STUDY. Decossas, K. M.; Koltun, S. P.; Eaves, P. H.; Spadaro, J. J.; Pollard, E. F.; and Patton, E. L. J. Am. Oil Chemists' Soc. 37: 574-76. 1960.

Methyl esters made directly from acidulated soapstocks of cottonseed, soybean, and corn oils by a process developed at the Southern Utilization Research and Development Division are being marketed as a high-energy fat additive for poultry and livestock feed. A flow sheet is given, together with descriptions of hypothetical plants with capacities of 15,000 to 60,000 lbs. of the acidulated soapstocks per 24 hours for the continuous production of up to 21 million pounds of methylated foots annually. The lowest manufacturing costs are realized for each plant when operating 24 hours a day, 250 days annually, averaging 5 days per week. For these optimum operations the estimated capital investment for the small plant is $223,000, and for the large plant $410,000. Manufacturing costs range from a high of 11.2¢ per pound of product at an annual production of 15.3 million pounds. The cost of the raw materials, although only 3.4¢ per pound of product and chiefly the cost of foots, is the largest single item of unit cost in producing methyl esters; and for the higher productions covered by this study, raw material costs account for more than one-half of total unit manufacturing cost.

2029. EFFECTS OF PROLONGED INTRAVENOUS ADMINISTRATION OF FAT EMULSIONS ON SERUM PROTEIN OF DOGS. Singleton, W. S.; Benerito, R. R.; Talluto, K. F.; Brown, M. L.; DiTrapani, L. L.; and White, J. L. Metabolism, Clin. and Exptl. 9: 956-65. 1960.

For extended periods 20 dogs received intravenous infusions of 30 to 50 ml./kg./day of two 15 percent cottonseed oil emulsions differing only in the emulsifying agent used (Lipomul-I. V. and SR, an emulsion developed at the Southern Utilization Research and Development Division). Serum protein fractions were determined by paper electrophoresis salt fractionation and in a few samples by ultracentrifugation. The major difference observed in sera following long-term infusions of the two emulsions was the magnitude of the decrease of serum albumin at dosages of 40 ml./kg./day about twice as much in dogs receiving Lipomul-I. V. as in the dogs receiving SR emulsions (decreases of 35 to 40 percent and 20 to 25 percent, respectively). In those dogs that did not survive the full course of infusions of either emulsion, the relative percentage of serum albumin progressively decreased by approximately 37 percent. The decrease in serum albumin for the dogs that survived the full course of infusions was only about half as great. Serum obtained during individual infusions was characterized by decreases in the relative percentage of the albumin fractions and in the beta and gamma fractions, indicating protein-lipid complexing. In the post-infusion periods, albumin concentration tended to return to the original values for a given dog, except during the final week of infusions. The decrease in serum albumin was not accompanied by a corresponding drop in total serum protein; these decreases appeared to be caused at least partly by formation of an albumin-lipid complex with changed mobility. At the dosage level of 40 mg./kg., formation of this complex appeared to be greater with Lipomul-I. V. than with the SR emulsion. It is concluded that the soya phosphatides used in Lipomul-I. V. are not as well tolerated as the polyethylene glycol stearate-TEM 4T in the SR emulsion.

2007. HYDROGENATION OF METHYL OLEATE IN SOLVENTS. Cousins, E. R.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 37: 435-38. 1960.

The hydrogenation of the oleic acid group was investigated to determine the effect of solvents on the reaction rate and the formation of positional and geometrical isomers. Methyl oleate, either alone or dissolved in one of several solvents, hexane, ethanol, n-butyl ether, or acetic acid, was hydrogenated to an iodine value of about 50 under atmospheric pressure and at 30° C. with palladium-on-carbon and the W-5 form of Raney nickel as catalysts. Hydrogenation with palladium catalyst, with or without solvents, produced 76.6 to 79.1 percent trans

bonds, based on the total double bonds. This is significantly greater than the 67 percent obtained previously. Hydrogenation products obtained with Raney nickel and solvents contained as little as 20.7 percent trans bonds at an iodine value of about 50. In two cases the trans bonds were equal to about one-third the positional isomers. Positional isomers formed extensively when the palladium catalyst was used. When the Raney nickel and solvents were used, large proportions of double bonds were found in the original 9-position.

2005. FIND MOLDING DIFFICULTY IN TEST COATINGS USING COCOA BUTTER-LIKE FAT. Feuge, R.O. Candy Ind. & Confectioners J. 115(4): 11-13, 30. 1960.

The article describes some of the work on confectionery fats underway at the Southern Regional Research Laboratory. The preparation of several cocoa butter-like fats prepared from completely hydrogenated cottonseed oil and olive oil is discussed. The best of the cocoa butter-like fats possessed the short melting range, hardness at room temperature, and other desirable characteristics of cocoa butter. Unlike cocoa butter, none of the fats contracted to the desired degree on molding. Also, they possessed a relatively high viscosity when worked at the temperatures ordinarily used with cocoa butter. The preparation and characteristics of slab oils, which can be classed as another type of confectionery fat, also are discussed. The performance of an experimental slab oil consisting essentially of dibutyropalmitins and dibutyrostearins is described.

1998. COMPARATIVE EFFECTS OF ORAL AND INTRAVENOUS ADMINISTRATION OF FAT EMULSION ON SERA FRACTIONS OF DOGS. Benerito, R.R.; Talluto, K.F.; White, J.L.; and Singleton, W.S. Metabolism, Clin. and Exptl. 9: 769-77. 1960.

Paper electrophoresis was applied to dog sera to determine quantitative and qualitative changes in the sera fractions after oral and intravenous administration of the same fat emulsion, and to detect any difference in the responses produced by a phosphatide and a nonphosphatide emulsifier. Single adminstration of 10 ml./kg. were employed in all cases. A pre-alpha-lipoprotein fraction was observed after administration of fat emulsions either orally or intravenously. The concentrations of this fraction were low except after intravenous administration of the nonphosphatide emulsion, when the concentration and mobility of pre-alpha-lipoprotein were greatly increased. Transformation of the lipoprotein fraction normally migrating as alpha-lipoprotein was responsible for this increase in pre-alpha-lipoprotein. This phenomenon was the most significant difference between the two emulsions and between the two routes of administration. Slight changes in protein content and relative proportions of protein fractions were common with both emulsions and both routes of administration but could not be distinguished from normal variation.

1956. ADDITIONAL PHYSICAL PROPERTIES OF DIGLYCERIDE ESTERS OF SUCCINIC AND ADIPIC ACIDS. Feuge, R.O.; and Ward, T.L. J. Am. Oil Chemists' Soc. 37: 291-94. 1960.

Diglycerides of the fat-forming acids yield, on esterification with succinic, adipic, and other short-chain dibasic acids, a potentially useful series of compounds ranging from hard, high-melting waxes to viscous oils that will not crystallize. A number of the properties of these compounds were determined in earlier investigations. In the present investigation additional properties of the 1,3-diolein and 1,3-distearin esters of succinic and adipic acids were determined. Surface and interfacial tensions were measured and found to be similar to those of cottonseed oil. The smoke points also were found to be similar to that of cottonseed oil. The ability of the compounds to thicken cottonseed oil was measured and found to be somewhat better than that of highly hydrogenated cottonseed oil at levels above about 12 percent, and the mixtures were relatively resistant to fat leakage. In hardness the distearin esters of succinic and adipic acid were comparable to carnauba wax and were over twice as hard as highly hydrogenated cottonseed oil. Permeability to water vapor was greater than that of highly hydrogenated cottonseed oil and carnauba wax and about equal to that of cocoa butter.

1912. FACTORS AFFECTING OIL PARTICLE SIZE IN THE FREEZING AND THAWING OF FAT EMULSIONS. Singleton, W. S.; Benerito, R. R.; and White, J. L. J. Am. Oil Chemists' Soc. 37: 88-92. 1960.

A film of Lipomul-I. V. can be frozen and thawed repeatedly with no apparent increase in the size of the dispersed oil particles. The rate at which a full, frozen bottle of Lipomul-I. V. was thawed appeared to be more important than the rate of freezing in preventing an increase in oil particle size. Frozen Lipomul-I. V. was acceptable in particle size only when thawed over a period of 20 hours, irrespective of the rate of freezing. The optimum thawing rate of a frozen emulsion varies with the melting point of the oil, provided the melting point of the oil is lower than the melting point of water, and with the type of emulsifying system used. Cottonseed oils with melting points of 10° and 49° C., respectively, increased in particle size with three different rates of thaw. Oils with low melting point are less affected by the emulsifier system and the solid-liquid relationships existing in the oil phase at various temperatures. Safflower oil emulsions are acceptable in particle size with at least two different emulsifier systems after freezing and thawing at slow, moderate, and rapid rates. Emulsions can be made from fractionated cottonseed oil, with either a phosphatide or nonphosphatide emulsifier system not only to equal in particle size emulsions made with safflower oil but to thaw in the same manner as safflower oil. An oil content of 30 percent in an emulsion did not prevent an increase in oil particle size after a freezing-thawing cycle. Such an emulsion creamed on thawing.

1908. PERMEABILITY OF SOME FAT PRODUCTS TO MOISTURE. Landmann, Werner; Lovegren, N. V.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 37: 1-4. 1960.

Films of cocoa butter, highly hydrogenated cottonseed oil, mixtures of highly hydrogenated cottonseed oil and cottonseed oil, chocolate liquor, and sweet milk chocolate were prepared and their permeability to water vapor determined by the cup method. The permeability constant was calculated in terms of grams of water diffusing through a centimeter cube in one second under a vapor pressure gradient of one millimeter of mercury across the cube. The permeability constant for cocoa butter at room temperature varied from 5.8×10^{-12}. Permeability constants for the highly hydrogenated cottonseed oil and the cocoa butter, under comparable conditions at room temperature, were approximately 1.3×10^{-12}, respectively. From data obtained with cocoa butter it was concluded that the permeability constant increased with moderate increases in film thickness. Polymorphism had a large effect on permeability; approximately a 15-fold difference was found between quickly chilled and tempered films of cocoa butter at 3°C. (37.4°F.). The percentage of liquid in the fat had a large effect on permeability. The increasing of the percentage of liquid cottonseed oil in a highly hydrogenated cottonseed oil from 0 to 40 percent increased the permeability constant from 1.3×10^{-12}. The permeability of chocolate liquor and sweet chocolate at room temperature increased greatly when the relative humidity on the wet side of the films was increased to 100 percent. The nonfat components absorbed enough moisture to impair the structure of the film.

1878. (RECENT DEVELOPMENTS IN THE MODIFICATION OF FATS). Feuge, R. O. Candy Ind. and Confectioners J. 113: 2, 7, 12, 62; and Candy Ind. and Confectioners J. 113: 3, 32, 33, 36, 37. 1959. Published in two parts under titles: "Lab Studies Show Way to New Candy Fats" and "Lab Can Combine Selected Characteristics."

The manuscript was submitted under the title "Recent Developments in the Modification of Fats." The editor of the Journal divided the manuscript into two parts entitled "Lab Studies Show Way to New Candy Fats" and "Lab Can Combine Selected Characteristics." The first article describes in terminology understandable to nonchemists, the nature of fats, changes that can be made in their structure, and current practices in the fractionation of fats to produce products having more desirable characteristics. The second article describes other

modification procedures: rearrangement and directed rearrangement, esterification, and hydrogenation. Probable future developments are predicted.

1872. POLYMERIC FATS FROM STEARIC, OLEIC, AND SHORT-CHAIN DIBASIC ACIDS. Feuge, R. O.; and Gros, A. T. Ind. Eng. Chem. 51: 1019-22. 1959.

A need exists in the food industry for highly viscous oils and low-melting fats of exceptional resistance to oxidation. Such products can be prepared by polymerizing with the aid of short-chain dibasic acids the mono- and diglycerides derived from ordinary fats. Products prepared by the acylation of 1-mono-olein with one of the dibasic acids, fumaric, succinic, or adipic, possessed viscosities up to 325 centistokes at 50°C. and did not crystallize at temperatures below 0°C. Similar products prepared with stearic acid instead of oleic acid possessed viscosities up to 2,360 centistokes at 50°C. The melting points ranged between 35.5 and 58.2°C. When the glycerol employed in the preparation was used in excess of that required to react with the carboxyl groups present, the viscosity and average molecular weight of the product decreased, and the melting point increased. The average molecular weight of the products never exceeded 1,700. Theoretical considerations and experimental data indicated that no compounds having molecular weights above about three or four thousand were prepared. No experimental data are available on the physiological behavior of these compounds on ingestion.

1870. NEW FAT PRODUCTS: GLYCERIDE ESTERS OF ADIPIC ACID. Ward, T. L.; Gros, A. T.; and Feuge, R. O. J. Am. Oil. Chemists' Soc. 36: 667-71. 1959.

Six new glyceride esters of adipic acid were prepared, and their properties determined. By the use of acetals and trityl groups, the number, type, and positions of the fatty acid groups in the glycerol moiety were varied systematically. The reaction products were purified, their purities were established, and their melting points, viscosities, densities, and refractive indices were determined.

1865. EFFECT OF COMPOSITION AND POLYMORPHIC FORM ON THE HARDNESS OF FATS. Feuge, R. O.; and Guice, W. A. J. Am. Oil Chemists' Soc: 36: 531-34. 1959.

Hardness is an important index to the performance of confectionery and other fats. Using an instrument and a technique that are essentially a modification of those used in the Brinell test applied to metals, the effect of composition and polymorphic form on the hardness of fats was investigated. The hardness of a given sample of fat was influenced by the degree of tempering to which the samples had been subjected. Hardness increased as the components of a fat were converted to higher-melting polymorphs. However, the hardest specimens were not obtained with the highest tempering temperatures. Presumably, the use of too high a temperature in tempering melted some of the lower-melting polymorphs and allowed them to resolidify in large crystals producing a softer matrix. Adding progressively larger amounts of one fat to another generally increased or decreased the hardness of the mixture in a more or less uniform manner. Adding small amounts of liquid oil to a hard fat greatly decreased the hardness index. Apparently, the hardness index of a given fat decreases as the crystal size increases. It is believed that fats containing a sizable proportion of liquid component will become softer on prolonged storage because the presence of the liquid component makes possible a gradual increase in crystal size.

1780. SEES COCOA BUTTER DUPLICATION. (Confectionery Fats of the Future). Feuge, R. O. Candy Ind. and Confectioners' J. 112(12): 14-15, 131. 1959.

Changes in the fat and oil industry during the last 50 years are described briefly. Using these changes as a yardstick and considering the fact that the fat and oil industry is becoming more

inclined to increase the complexity of processing operations in order to produce special, tailored products, future developments in the way of new fat products are suggested. The modification of domestic oil to duplicate cocoa butter, the most prized confectionery fat, is shown to be probable, as is the development of confectionery fats that are resistant to bloom and that will not soften at summer temperatures, yet will remain palatable. The development of superior, edible oils for use as a slab dressings and as polishing oils in the manufacturing of confections is predicted, as well as the development of new and better food emulsifiers.

1770. MOISTURE TRANSMISSION THROUGH FATS. (Condensation). Feuge, R. O.; Mfg. Confectioner 39(6): 79-80. 1959. (Republished: Speeches and Proc. Ann. Prod. Conf. Penn. Mfg. Confectioners' Assoc. 13: Sect. XIV. 9 pp. 1959.)

The permeability of a number of fat products to water vapor was measured by the cup method. Permeability was calculated in terms of an index, taking into consideration the surface area and thickness of the film of fat, the vapor pressure difference across the film, and the amount of water transferred per unit of time. Permeability indices for the fat products were found to range from 2.44 to 2,340. The latter value was obtained for a sweet milk chocolate tested under a relative humidity so high that the absorbed moisture damaged the structure of the film. By comparison, the approximate permeability indices found by others for several nonfat products are: cellulose acetate, 850; nylon, 55; polyethylene, 1.5; and paraffin, 0.3. Paraffin apparently is the least permeable of all organic compounds. The passage of moisture through films of fat under a given set of conditions was generally found to be relatively unaffected by moderate variations in the thickness of the film. The amount of moisture passing through a film under a given difference in vapor pressure sometimes was affected greatly by the relative humidities employed in the tests. Increasing the amount of liquid oil in a fat from 0 percent to 20 percent greatly increased the permeability index. The manner in which a fat was solidified and its subsequent heat treatment had a large effect on the permeability index. Seeding during solidification and tempering reduced the permeability to water vapor.

1769. METHYL ESTERS DIRECTLY FROM ACIDULATED SOAPSTOCK. Eaves, P. H.; Spadaro, J. J.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 36: 230-34. 1959. (Republished: PREPARATION DIRECT D'ESTERS METHYLIQUES A PARTIR DE PATES DE NEUTRALISATION ACIDIFICES).

A method has been developed for utilizing low-cost acidulated refinery soapstock directly as raw material for producing the mixed methyl esters of cottonseed oil fatty acids. The conditions determined as optimum for producing the esters were as follows: temperature, 110° C. to 120° C.; pressure, 150 p. s. i. g.; reaction time, 10 to 15 min.; catalyst, acidic, proportioned as 3 percent to 5 percent of the fatty acids of the foots; and ratio of reactants, 5 moles of methanol to 1 of fatty acids. Processing acidulated foots containing 12.4 percent of neutral oil at the foregoing conditions resulted in esterifying 83 percent to 86 percent of the fatty acids. Reprocessing the same material after dehydrating it and restoring the initial methanol to fatty acids ratio increased the percentage of fatty acids esterified by about 5 percent. Following the same procedure but using foots that had been hydrolyzed to reduce their neutral oil content to less than 1 percent, one-pass processing esterified 91 percent to 92 percent of the fatty acids, with a second pass increasing the percentage of fatty acids esterified to 96 percent or 97 percent.

1763. ADDITION OF HYDROGENATED FATS TO CHOCOLATE TO IMPROVE HEAT RESISTANCE. Guice, W. A.; Lovegren, N. V.; Feuge, R. O.; and Cosler, H. B. J. Am. Oil Chemists' Soc. 36: 4-8. 1959.

An investigation was made of the possibility of fortifying chocolate better to withstand summer temperatures by adding relatively small proportions of completely hydrogenated cottonseed

oil and similar products. Proportions of hydrogenated oils up to 10 percent, based on the weight of cocoa fat, do not affect mouthing quality significantly, yet they impart marked rigidity to the chocolate mass at temperatures at which cocoa fat softens and melts. Because the hard fat is practically insoluble in cocoa fat at the melting point of the latter, the hard fat, under the conditions adopted, does not alter the melting characteristics of the cocoa fat, and the short softening range of the cocoa fat is retained. The addition of the completely hydrogenated oil greatly retards the rate of fat leakage from chocolate at temperatures of about 36° C. (97° F.). The addition of small amounts of very hard fats to chocolate should be useful in improving the performance of molded bars; however, if the modified chocolate is to be used for coating confections, the increase in viscosity that results on the addition of the hard fat presents a serious disadvantage.

1691. PREPARATION OF PURE PALMITIC ACID AND A BYPRODUCT PLASTICIZER FROM COTTONSEED OIL. Magne, F. C.; Mod, R. R.; and Skau, E. L. J. Am. Oil Chemists' Soc. 35: 477-79. 1958.

A laboratory process has been developed that permits the isolation of a highly purified palmitic acid from cottonseed acids through the recrystallization of its cyclohexylamine salt from acetone and subsequent regeneration of the acid. The byproduct, predominantly cyclohexylamine salts of the unsaturated fatty acids, can be modified by conversion first to morpholine salts and then to morpholides that have shown promise as vinyl plasticizers. The morpholine salts can be obtained as a direct byproduct by the use of an appropriate mixture of cyclohexylamine and morpholine with the composite acid. By using an amount of cyclohexylamine closely equivalent to the saturated fatty acid content and sufficient morpholine to make up the residual neutralization equivalent of the composite cottonseed acids, the saturated acids can be isolated as cyclohexylamine salts. The stripped mother liquor will consist essentially of the morpholine salts of the unsaturated acids. Pure palmitic acid is obtained by recrystallization of the cyclohexylamine salts. The complete process is applicable to fatty acids from other saturated acids closely paralleling that in cottonseed oil. The process is not operable with partly hydrogenated oils.

1688. 1, 3-DIOLEIN AND 1, 3-DISTEARIN ESTERS OF FUMARIC, SUCCINIC, AND ADIPIC ACIDS. Feuge, R. O.; and Ward, T. L. J. Am. Chem. Soc. 80: 6338-41. 1958.

To obtain information on such dibasic acid glycerides, the 1, 3-diolein and 1, 3-distearin esters of fumaric, succinic, and adipic acids were prepared by the reaction between the 1, 3-diglycerides and the acid chlorides of the dibasic acids in the presence of pyridine or quinoline. Special precautions were taken in most instances to reduce the amount of side reaction. The reaction products were purified and a number of their physical properties established. The melting points of the highest melting polymorphic form of the distearin-containing compounds ranged from 82. 1 to 89. 9° C. Either the diolein-containing compounds could not be obtained in a crystalline form or crystallization was incomplete, hence the melting points of these compounds were not determined. It was established that the form obtained on quick chilling and the highest melting form of the distearin-containing compounds crystallized in a single-chain-length structure. The short spacings for the forms obtained on quick chilling were always equal to the short spacing characteristics of the alpha form of tristearin. The compounds crystallized as long needles, and in some instances clumps of crystals were obtained that looked like asbestos. All of the diglyceride esters of the dibasic acids were quite viscous when compared with cottonseed oil at the same temperatures. The viscosity of the diolein-containing compounds ranged from 77. 00 to 92. 00 centistokes at 38. 8° C. The viscosity of the distearin-containing compounds ranged from 11. 42 to 18. 93 centistokes at 98. 8° C. On the basis of these data, diglyceride esters of the dibasic acids can be said to be approximately twice as viscous as cottonseed oil.

1685. LECITHIN IN OIL-IN-WATER EMULSIONS. Yeadon, D. A.; Goldblatt, L. A.; and Altschul, A. M. J. Am. Oil Chemists' Soc. 35: 435-38. 1958.

The use of purified phosphatide in fat emulsions for intravenous alimentation was investigated. Lecithin isolated from egg yolk and purified from alumina and silicic-acid chromatography was was analyzed by chromatographic strip techniques as a one-component material. Highly purified lecithin is an inefficient emulsifier. Moreover, emulsions containing highly purified lecithin are heat-sensitive. An emulsion physical stability test was developed to evaluate emulsifier formulations containing purified phosphatides for use with small amounts of emulsions (approximately 50 g.). By this procedure, a number of substances were tested as additives to enhance the purified lecithin's emulsifying power. No combination was found to be as effective an emulsifier as the natural soybean phosphatide used as a control. These observations indicate that pure phosphatides are inefficient emulsifiers and that those phosphatide preparations possessing good emulsifying characteristics are presumably mixtures or complexes of the phosphatides with other substances.

1683. AN INSTRUMENT FOR MEASURING THE HARDNESS OF FATS AND WAXES. Lovegren, N. V.; Guice, W. A.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 35: 327-31.

Instruments currently used to measure hardness of fats and waxes (not consistency) are relatively inaccurate and give results based on an arbitrary scale. A new instrument and technique have been devised, essentially an adaptation of the Brinell hardness test used for metals and alloys. In determining the hardness of a fat or wax, a perfectly round steel ball having a diameter as small as 0.1250 in. or as large as 0.5000 in. is pressed with a force of 200 g. to about 6 kg. onto the surface being tested. The hardness index, in terms of kilograms per square centimeter, is determined from the slight impression produced. The index is relatively independent of ball size, test load, and other test conditions provided these are confined to certain ranges. Hardness indices have been determined for products ranging from poorly tempered cocoa butter to rosin. Differences in hardness observed varied over 1,500-fold. The hardness of cocoa butter varies over sevenfold, depending on thermal history.

1628. SOME N-DISUBSTITUTED AMIDES OF LONG-CHAIN FATTY ACIDS AS VINYL PLASTICIZERS. Magne, F. C.; Mod, R. R.; and Skau, E. L. Ind. Eng. Chem. 50: 617-18. 1958.

Preliminary tests of morpholides of certain fatty acids as plasticizers show favorable efficiency, compatibility, and brittle point, but somewhat inferior heat resistance. Preparation of amides, plasticizer evaluation procedure, and summation of results are included. A table of plasticizing characteristics of N-disubstituted amides of fatty acids from cottonseed and peanut oils is given.

1623. THE PRESENT STATUS OF ACETOGLYCERIDES. Alfin-Slater, R. B.; Coleman, R. D.; Feuge, R. O.; and Altschul, A. M. J. Am. Oil Chemists' Soc. 35: 122-27. 1958.

Acetoglycerides can be prepared readily by several procedures, which are described. Fairly extensive investigations of the physiological behavior of acetoglycerides have been made. It is concluded from work reported to date that acetoglycerides have no deleterious effect on growth or on reproduction and that the mode of digestion of aceto-oleins and aceto-stearins does not differ appreciably from that of the corresponding natural fats. The acetyl group appears to be hydrolyzed and absorbed quickly and completely; no acetate has been demonstrated in the feces of acetoglyceride-fed rats. The rate of absorption of aceto-oleins exceeds that of aceto-stearins, but that for the latter is not unlike that of a commercial shortening. As to overall

digestibility, that of aceto-oleins, fed at levels up to 50 percent of the diet, appears to be equal to that of the corresponding normal triglycerides, whereas the digestibility of aceto-stearins, when measured in animals prefed or simultaneously fed sufficient unsaturated fat to preclude essential fatty acid deficiency, is markedly better than the absorption of the normal saturated glycerides.

1617. FAT EMULSIONS. EFFECT OF POLYOXYETHYLENE AND ALKYL CONTENT OF EMULSIFIERS ON STABILITY TO STERILIZATION. Singleton, W. S.; White, J. L.; Benerito, R. R.; and Talluto, K. F. J. Am. Oil Chemists' Soc. 35: 265-70. 1958.

When a given system of emulsifiers of known composition and molecular weight is employed, in order for fat emulsions of 15 percent oil content to be stable to the heat required for sterilization, the emulsifiers must meet definite requirements. Minimum weight percentage of total lipophilic groups should be 0.29 percent; minimum weight percentage of total hydrophilic groups should be 0.57 percent; the polyoxyethylene groups of emulsifiers containing such groups should account for at least 70 percent of the mole weight of these emulsifiers; the lipophilic/hydrophilic ratio should be about 0.5. The minimum content of lipophilic and hydrophilic groups was an additive total of these groups as provided by the complete emulsifying system whether composed of individual or multiple emulsifying agents and whether of similar or dissimilar functional groups. With the emulsifiers used, an acyl group greater in length than 12 carbon atoms was necessary to prepare cottonseed oil emulsions that were stable to heat. Resistence of emulsions to breakage by mechanical shock was imparted by a rather large amount of lipophilic groups, so that the lipophilic/hydrophilic ratio was 1 or over.

1615. DYEING METHOD FOR QUANTITATIVE DETERMINATION OF LIPIDS AND LIPOPROTEINS DIRECTLY ON FILTER PAPER. Talluto, K. F.; Benerito, R. R.; and Singleton, W. S. Anal. Chem. 30: 1059-62. 1958.

A quantitative dyeing technique was required to measure accurately and rapidly the changes in serum lipid fractions after the administration of intravenous fat emulsions. The use of a 50 percent aqueous solution of diacetin saturated with Oil Red O dye at 25° C. prevents the loss of lipids from the paper strips into the dye bath, as usually occurs in alcoholic solutions of the dye at 25° C., and results in a linear relationship between dye uptake and lipid concentration for various classes of lipids. Direct scanning of the dyed strips by the method of transmission densitometry results in a linear relationship between integrated area and the concentration of a given lipid. Due to the differences in solubilities of the oil-soluble dye in various classes of lipids, there are differences in the partition coefficients of the dye between the dye bath and the various classes of lipids. However, within a given class, the variation with alkyl group is small; for example, the dye uptake of tristearin varies little from the dye uptake of an equal quantity of triolein.

1614. COCOA BUTTER-LIKE FATS FROM DOMESTIC OILS. Feuge, R. O.; Lovegren, N. V.; and Cosler, H. B. J. Am. Oil Chemists' Soc. 35: 194-99. 1958.

Technical considerations indicate that cocoa butter-like mixtures can be prepared readily by the esterification of mixtures of oleic, palmitic, and stearic acids, or the interesterification of their glycerides, followed by the fractional crystallization of the reactant products. Three cocoa butter-like fractions were prepared by the indicated procedures. One consisted essentially of oleopalmitostearins, the second of oleodistearins, and the third of oleodipalmitins. On the basis of softening point curves, the oleopalmitostearin product was the next, and the oleodipalmitin product was least compatible. When mixed with cocoa butter, all three of the products produced consistency versus temperature curves whose shape closely resembled that of cocoa butter. All of the mixtures softened over a short temperature interval though the actual temperature at which mixtures softened varied. The several products are believed to be satisfactory cocoa butter replacements. Another cocoa butter-like fat was prepared by

the interesterification of 70 parts of completely hudrogenated cottonseed oil and 30 parts of olive oil and the subsequent fractionation of the reaction product.

1541. RAPID DYEING METHODS FOR THE QUANTITATIVE ANALYSIS OF SERUM PROTEINS BY PAPER ELECTROPHORESIS. Formusa, K. M.; Benerito, R. R.; Singleton, W. S.; and White, J. L. Anal. Chem. 29: 1816-20. 1957.

A quantitative dyeing technique was needed to measure accurately and rapidly the changes in serum protein fractions after the administration of intravenous fat emulsions. Under rigidly standardized conditions, the amount of dye uptake is proportional to the concentration of protein nitrogen, regardless of the type of serum protein. Direct scanning of the dyed and oiled strips by the method of transmission densitometry results in a linear variation of integrated area with the concentration of protein nitrogen. The method can be used to measure concentrations of serum proteins in the presence of fat emulsions.

1525. THE COMPOSITION OF BLOOM FAT IN CHOCOLATE. Cerbulis, J.; Clay, C.; and Mack, C. H. J. Am. Oil Chemists' Soc. 34: 533-37. 1957.

The composition of the fat that has bloomed in chocolate has been investigated. There is evidence that the type of center that is coated with chocolate has no effect upon the fatty-acid composition of the bloom fat. The rate of blooming, however, was found to be affected by the center. The theory that the oils in coated nuts migrated to the chocolate surface to cause fat bloom has been disproved. Comparison of the fatty acids present in the bloom fat with those in the coating fat show a decrease in the unsaturated acids and an increase in the saturated acids. There is evidence that hydroxyl-containing oxidation products of the fatty materials in the chocolate are not present in the migrated fat.

1524. COMPOSITION OF ACIDULATED COTTONSEED SOAPSTOCKS AS INFLUENCED BY COMMERCIAL METHODS OF PROCESSING SEED AND OIL. Stansbury, M. F.; Cirino, V. O.; and Pastor, H. P. J. Am. Oil Chemists' Soc. 34: 539-44. 1957.

Ninety-nine acidulated soapstocks have been analyzed to obtain compositional data needed for efficient utilization of these materials and for their evaluation in commercial trading. Most of the compositional characteristics of the samples were quite variable. The soapstocks from the direct solvent-extracted oils contained less total fatty acids and more oxidized fatty acids than those from the other types of crude oils. High neutral oil contents were observed for the soapstocks from hydraulic oils that had been batch refined and from high-speed screw-pressed oils that had been centrifugally refined. Gossypol was one of the most variable constituents. The samples from the direct-extracted and high-speed screw-pressed oils were comparatively high in gossypol content, whereas most of those from hydraulic oils were quite low. Unsaponifiable matter did not vary appreciably. The samples from the direct-extracted and high-speed screw-pressed oils were comparatively high in gossypol content, whereas most of those from hydraulic oils were quite low. The samples from direct-extracted and high-speed screw-pressed oils also contained greater amounts of phosphatides than did the other samples. The composition of the unoxidized (total) fatty acids from the soapstocks was in the same general range usually found for cottonseed oils, with the exception that oleic acid contents averaged slightly lower and saturated acids somewhat higher.

1473. PHYSICAL PROPERTIES OF ACETO- AND BUTYRO-OLEINS, MONO-OLEIN, AND DIOLEIN. Gros, A. T.; and Feuge, R. O. J. Am. Oil. Chemists' Soc. 34: 239-44. 1957.

Certain diglycerides and triglycerides containing both long- and short-chain fatty acids are liquid at low temperatures and exhibit unique properties. Four of these compounds, 1-aceto-3-olein, 1,2-diaceto-3-olein, 1-butyro-3-olein, and 1,2-dibutyro-3-olein, were prepared and some of their physical properties were determined. For purposes of comparison, data on

1-mono-olein and 1,3-diolein were obtained. Melting points of the highest melting forms ranged from -29° C. for the 1,2-dibutyro-3-olein to 32° C. for the 1-mono-olein. Melting points for the lower melting polymorphic forms of some compounds were not established definitely because of limitations of existing equipment. Expansibilities in the liquid state ranged from 0.000812 ml./g./°C. for the 1-mono-olein to 0.000912 ml./g./°C. for the 1,2-diaceto-3-olein. Specific volumes in the liquid state at 35° C. ranged from 1.0392 ml./g. for the 1,2-diaceto-3-olein to 1.0955 ml./g. for the 1,3-diolein.

1470. BROWNING IN PHOSPHATIDE-CONTAINING FAT EMULSIONS. Benerito, R.R.; Formusa, K.M.; White, J.L.; and Singleton, W.S. Proc. Soc. Exptl. Biol. Med. 94: 47-51. 1957.

Phosphatide-containing fat emulsions for intravenous alimentation frequently develop a colloidal dispersion of a brownish material that is not observed with emulsions made with other emulsifiers. This investigation gives experimental evidence that at autoclaving temperature (121° C.) a type of browning occurs that involves reaction of dextrose with lecithins as well as with amino-groups of cephalins. Therefore, use of pure lecithin as an emulsifier in an oil emulsion containing dextrose in the water phase would not eliminate formation of the brown material. The complex browning reaction is responsible for formation of the colloidal material and might be an explanation of certain adverse physiological results occasionally experienced with the use of these emulsions.

1457. THE DESTRUCTION OF GOSSYPOL IN COTTONSEED OIL SOAPSTOCK BY A HEAT TREATMENT. Pominski, Joseph; and Pack, F.C. J. Am. Oil Chemists' Soc. 34: 299-301. 1957.

On the basis of information developed in the laboratory, a continuous pilot-plant apparatus was designed to process approximately 0.25 gallons, or 1.88 pounds of raw alkaline soapstock per hour at temperatures of 212° to 215° C. and pressures of 290 to 300 pounds per square inch. The apparatus was operated successfully at rates of feed ranging from 1.89 to 5.51 pounds per hour. The data show that cottonseed oil soapstock can be heat treated continuously so that both the free and total gossypol content as measured by the ρ-anisidine method are reduced to values as low as 0.06 percent. Recycling the treated soapstock through the apparatus will lower the total gossypol content to 0.003 percent.

1392. PROGRESS IN A NEW AREA OF CANDY RESEARCH. Altschul, A.M. Natl. Confectioners Assoc., Rept. 73rd Ann. Convention, 1956: 19-21.

Research on the chemistry of fats and oils as a means of improving their properties for use in the making of candies is discussed. Knowledge of the general chemistry of fats and oils has already advanced sufficiently to justify belief that through study of the relationship between changes in composition and physical properties of the fat, cocoa butter can be reproduced, or a similar fat produced that has specific properties more suitable for certain types of candies. For example, fats may be produced compatible with cocoa butter but with higher melting points for use in summer candies. Other physical properties, such as moisture transfer, compatibility, melting point, and polymorphic form, may also be improved through greater knowledge of the chemistry of fats.

1391. PERMEABILITY OF ACETOSTEARIN PRODUCTS TO CARBON DIOXIDE, OXYGEN, AND NITROGEN. Lovegren, N.V.; and Feuge, R.O. Agr. & Food Chem. 4: 634-38. 1956.

The acetostearins are modified fats that can be formed into wax-like films potentially useful as protective coatings. Permeability to carbon dioxide was measured by modification of the standard cup method, using Ascrite as the absorbent. An apparatus was developed that could

measure in about 8 hours the passage of a few hundredths of 1 ml. of gas through a film of acetostearin, 2-3/8 in. in diameter, into a sweep gas that was analyzed. Permeability of the acetostearins to carbon dioxide was less than that reported by other investigators for ethylcellulose and approximately the same as for polystyrene and polyethylene. Nylon and regenerated cellulose have lower permeabilities. Polystyrene and ethylcellulose are more permeable to oxygen and nitrogen than acetostearin; polyethylene has approximately the same permeability. Cellulose acetate, regenerated cellulose, and nylon are less permeable.

1390. FAT EMULSIONS. EFFECT OF HEAT ON SOLUBILITY OF HYDROPHILIC EMULSIFIERS. Benerito, R. R.; and Singleton, W. S. J. Am. Oil Chemists' Soc. 33: 364-67. 1956. (Republished: Am. Perfumer Aromat. 69(3): 37-40. 1957).

Oil-in-water emulsions for intravenous use must be steam-sterilized before use and after sterilization must possess a particle size below one micron. One of the factors that might influence the choice of emulsifiers for such an emulsion is water solubility at elevated temperatures. Calculations to determine the amount of emulsifier required under certain conditions to coat the oil droplets of an emulsion as a monomelecular film are presented.

1389. DILATOMETRIC PROPERTIES OF SOME BUTYROPALMITINS AND STEARINS AND ACETOPALMITINS. Feuge, R. O.; and Lovegren, N. V. J. Am. Oil Chemists' Soc. 33: 367-71. 1956.

Certain diglycerides and triglycerides containing both short- and long-chain fatty acids normally solidify to waxy solids and as such have a number of potential uses. Six of these compounds (1-butyro-3-palmitin; 1, 2-dibutyro-3-palmitin; 1-butyro-3- stearin; 1, 2-dibutyro-3-stearin; 1-aceto-3-palmitin; and 1, 2-diaceto-3-palmitin) have been prepared and some of their physical properties have been determined. Expansibilities in the solid and liquid states, melting dilations, densities, melting points, points of transition from one polymorphic form to another, and ease of tempering to the higher polymorphic forms were measured or calculated. Melting dilations for the highest melting form of each compound ranged from 0.612 ml. to 1.203 ml. per 10 grams. The 1, 2-dibutyro-3-palmitin changed very slowly to its high melting form (m. p., 10° C.). On the other hand, the 1-butyro-3-palmitin and 1-butyro-3-stearin quickly temper to the high melting forms.

1230. SOME PRELIMINARY INVESTIGATIONS DIRECTED TOWARD INCREASING THE UTILITY OF COTTONSEED SOAPSTOCK. Pack, F. C.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 32: 551-53. 1955.

Byproduct soapstock that results from the refining of cottonseed oil contains numerous diverse substances. Crude benzyl esters of cottonseed fatty acids that have potential utility in the field of secondary plasticizers have been prepared by the direct action of benzyl chloride on alkaline soapstock. Acidulated soapstock has recently found its way into the feed industry. The possibility of using substantial amounts of either acidulated or raw soapstock in feeds revives interest in a rapid and economical method for getting rid of gossypol. In these investigations gossypol in raw soapstock was destroyed by simple heat treatment at temperatures above 200° C.

1219. DEVELOPING NEW PRODUCTS FROM COTTONSEED OIL AND FOOTS. Goldblatt, L. A. Cotton Gin & Oil Mill Press 56(15): 9, 34-36, 38. 1955.

Technological developments, such as hydrogenation and ester interchange, have resulted in greatly increased interconvertibility of oils. This leads to greater price stability and improved opportunities for industrial utilization. Considerable attention is being given to a search for new products from cottonseed oil and foots through chemical modification. One group of these is the acetoglycerides, which may take the form of a flexible, waxy solid, or an oil that remains liquid at very low temperatures. A convenient method of destroying

gossypol in cottonseed foots offers a possibility that foots may be used as an additive for animal feed. Cottonseed foots may also be treated with benzyl chloride to obtain benzyl oleate, an ester useful as a plasticizer for polyvinyl chloride, with the advantage of lowering the brittle point from -31° to -45° C. A number of other esters have been prepared from cottonseed foots, which may be useful for anything from lubricants to alkyd resins for paints and varnishes. Other products with interesting possibilities, which might be produced by suitable chemical modification, include dibasic acids such as azelaic, sebacic, and adipic acids, which are used extensively for production of plasticizers, resins, and fibers.

1172. SOME THERMAL PROPERTIES OF 1-MONOSTEARIN, 1-ACETO-3-STEARIN, AND 1,2-DIACETO-3-STEARIN. Ward, T. L.; Vicknair, E. J.; Singleton, W. S.; and Feuge, R. O. J. Phys. Chem. 59: 4-7. 1955.

Pure 1-monostearin, 1-aceto-3-stearin, and 1,2-diaceto-3-stearin have been examined calorimetrically over a temperature range of -73 to 100° C. The properties investigated were specific heat (Cp), heat of fusion, accumulated heat content, and heat of transition upon changing of polymorphic form.

1171. SOLUBILITY OF TRISTEARIN AND HYDROGENATED COTTONSEED OIL IN CERTAIN ACETO- AND BUTYROGLYCERIDES. Ward, T. L.; Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 32: 316-18. 1955.

Tristearin, triolein, 1,2-diaceto-3-olein, an aceto-olein product, and 1,2-dibutyro-3-olein were prepared. Also a commercial, refined, and bleached cottonseed oil was hydrogenated to obtain products having iodine values of 29.3 and 1.1. Solubility measurements were made for the mixtures tristearin-triolein, tristearin-1,2,diaceto-3-olein, tristearin-1,2-dibutyro-3-olein, and for hydrogenated cottonseed oils in the aceto-olein product and cottonseed oil. In mixtures containing from 0.18 percent to approximately 30 percent of tristearin, the solubility on a weight basis was unaffected by the nature of the oil. On a mole fraction basis the tristearin was most soluble in the triolein and least soluble in the 1,2-diaceto-3-olein. Hydrogenated cottonseed oil appeared to be about equally soluble, on a weight basis, in the aceto-olein product and cottonseed oil.

1167. ACETOGLYCERIDES--NEW FAT PRODUCTS OF POTENTIAL VALUE TO THE FOOD INDUSTRY. Feuge, R. O. Food Technol. 9: 314-18. 1955.

The preparation and properties of some unique fat and oil products obtained by substituting, in effect, acetic acid for some of the fatty acids ordinarily combined in fats and oils are described. By proper choice of starting materials and degree of acetylation these new-type fats and oils or acetoglycerides can be tailored within limits to possess specific properties. Insofar as potential food uses are concerned, the acetoglycerides may be divided into three classes: (1) nongreasy, plastic fats of relatively sharp melting points for use as protective coating materials; (2) low-melting fats having extraordinary resistance to rancidity; and (3) low-melting oils that possess good resistance to rancidity and remain either liquid or plastic at temperatures far below freezing. A number of possible uses for the various acetoglyceride products are described.

1166. SURFACE PHENOMENA OF FATS FOR PARENTERAL NUTRITION. Singleton, W. S. W. S.; and Benerito, R. R. J. Am. Oil Chemists' Soc. 32: 23-25. 1955.

Surface and interfacial tensions were measured on peanut, cottonseed, coconut, pecan, olive, and rice oils and on two synthetic fats. Surface tensions of all the oils were found to decrease with increasing temperature. The spread in values of the refined oils at 25° isotherm was about 4.5 dynes cm^{-1}, with coconut oil lowest in value and commercial peanut oil highest. The spread of values obtained for the interfacial tensions at 25° was considerably wider,

9 dynes cm^{-1}, and the order was not the same as it was with increasing surface tension values. Representative crude oils had much lower interfacial tensions than did the refined oils.

1165. PREPARATION AND PROPERTIES OF BETA-LACTYL-ALPHA, GAMMA-DIPALMITIN. Goldblatt, L. A.; Yeadon, D. A.; and Brown, Mona. J. Am. Chem. Soc. 77: 2477-79. 1955.

Lactyl glycerides have been under investigation as emulsifiable fats for intravenous alimentation. Beta-lactyl-alpha, gamma-dipalmitin was prepared by the reaction of alpha, gamma-dipalmitin with the benzyl ether of lactyl chloride and hydrogenolysis of the benzyl group. Ultraviolet spectra to 220mμ and infrared spectra from 2μ to 12μ of benzyllactic acid, the benzyl ether of lactyldipalmitin, and of lactyldipalmitin were obtained.

1101. CONSISTENCY OF FATS PLASTICIZED WITH ACETOGLYCERIDES. Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 31: 377-83. 1954.

Several natural oils, normally liquid, were modified by ester interchange or glycerolysis and acetylation to produce acetoglycerides. An aceto-olein product was also prepared from commercial oleic acid. These products, together with two similar products obtained from a commercial firm, were tested to determine their ability to plasticize fats. Margarine-like products of wide plastic range could be prepared by acetylating lard and cottonseed, soybean, and peanut oils to an acetyl content ranging from about 12 to 20 percent. Simple addition of acetoglycerides to commercial margarine oil does not result in any improvements. Addition of an acetoglyceride-hard fat mixture improved the plastic ranges, provided addition was at a level of about 50 percent. A mixture of hard fat and peanut oil acetylated to a limited degree was found to have a much longer plastic range than did commercial margarine oil, and the consistency of the mixture was relatively constant over the temperature range of 15° to 30° C. Small amounts (1 to 6 percent) of aceto-olein product incorporated in highly hydrogenated cottonseed oil were effective in reducing the hardness of the latter when it was cast in block form.

1026. SOME PHYSICAL PROPERTIES OF THE POLYMORPHIC FORMS OF 1,2-DIACETO-3-STEARIN AND 1-ACETO-3-STEARIN. Vicknair, E. J.; Singleton, W. S.; and Feuge, R. O. J. Phys. Chem. 58: 64-66. 1954.

It was discovered here that fatty products containing the triglyceride (1,2-diaceto-3-stearin) and the diglycleride (1-aceto-3-stearin) are nongreasy solids relatively highly plastic and stretchable. These products appear to have practical uses. The two glycerides were prepared, purified, and examined dilatometrically over the temperature range of -17 to 60° C. Expansibilities, melting dilations, and temperatures at which polymorphic transitions occurred were determined from the dilatometric curves obtained. X-ray data for all polymorphic forms of each glyceride were obtained, and crystal spacings were calculated. Photomicrographs of two forms of each glyceride were made.

1022. FOOD COATINGS, PERMEABILITY OF ACETOSTEARIN PRODUCTS TO WATER VAPOR. Lovegren, N. V.; and Feuge, R. O. J. Agri. Food Chem. 2: 558-63. 1954.

One of the properties of acetostearin products being evaluated is permeability to moisture. A modification of the standard cup method was used, and measurements were made at various temperatures and relative humidities using films of different compositions and thicknesses. Permeability constants calculated for the fat films were not independent of film thickness nor concentration of water vapor. Permeability of acetostearin films to water vapor is less than that of cellulose acetate and slightly greater than that of nylon, ethylcellulose, and polystyrene. Cellophane and paraffin wax have significantly smaller permeabilities.

949. THE ACETOGLYCERIDES AS PLASTICIZERS FOR VINYL RESIN. Magne, F. C.; and Mod, R. R. J. Am. Oil Chemists' Soc. 30: 269-71. 1953.

Acetoglycerides can be used as secondary plasticizers to improve some of the plasticizing qualities of tricresyl phosphate and Santicizer 141, and as extenders for dioctyl sebacate. More work will be needed to confirm the effect of unsaturation, polyunsaturation, and degree of acetylation on plasticizing characteristics of acetoglycerides. Then their potentialities as plasticizers for vinyl chloride resins and other resins can be determined more precisely.

946. MODIFICATION OF VEGETABLE OILS. XIV. PROPERTIES OF ACETO-OLEINS. Feuge, R. O.; Gros, A. T.; and Vicknair, E. J. J. Am. Oil Chemists' Soc. 30: 320-25. 1953.

Objectives were to determine the properties of 1,2-diaceto-3-olein, triolein, and a mixture of aceto-oleins; to determine the properties of acetylated oils prepared by interesterification with triacetin or by glycerolysis followed by acetylation; and to examine the plastic properties of mixtures of highly hydrogenated cottonseed oil and aceto-containing glycerides. These mixtures, when compared with partly hydrogenated oil, butterfat, or a mixture of cottonseed oil and hydrogenated cottonseed oil, were softer below room temperature and firmer above room temperature. A margarine-like product containing 79 percent of aceto-olein and 18.5 percent of highly hydrogenated cottonseed oil had a consistency practically constant over the temperature range of -15° to 49° C. (5° to 120° F.).

737. MODIFICATION OF VEGETABLE OILS. XII. PLASTICITY OF SOME ACETO DERIVATIVES OF MONOSTEARIN. Feuge, R. O.; Vicknair, E. J.; and Lovegren, N. V. J. Am. Oil Chemists' Soc. 29: 11-14. 1952.

Acetostearins were prepared by reacting various proportions of acetic anhydride with a monostearin of 99.2 percent purity, a commercially available monostearin containing 91.5 percent monoglycerides, and a technical grade monostearin containing 61 percent monoglycerides. The last product was prepared from a completely hydrogenated cottonseed oil. All the acetostearins tested were nongreasy solids at room temperature, yet were highly flexible. At 22° C. most of them could be stretched more than 800 percent under the test conditions. Some had good flexibility even at 4°. Depending on the purity of the monostearin used and the degree of acetylation, some products melted completely below body temperature (37°) and others at temperatures up to 60°. The temperature interval over which melting occurred was usually very short (1° to 30°); however, products made from technical grade monostearin melted over the range 10° to 20° C.

597. A STUDY OF THE STABILIZATION OF FATS INCORPORATED IN CANDY. Robinson, H. M. Food Technol. 5(1): 20-24. 1951.

A method of accelerating fat oxidation by the use of traces of copper, added as copper stearate, has been applied in studying the deterioration of fats in candies. The method was applied to test samples of butter and of hardened vegetable oils for peroxide values and free fatty acids after storage at 86° F. in air ovens. The fats in butter creams and butter caramels were tested in the same way. A series of antioxidants was tested in the fats alone, in the fats with added copper, and with these same fats incorporated in candies. Hardened vegetable oils showed very little oxidative rancidity even with added copper, so antioxidants were of little value. Butter with added copper showed definite peroxide values in a short period and this period can be materially lengthened by the use of antioxidants. Final evaluation of the results awaits the completion of the tests on candies without the added accelerator.

585. TAILOR MADE FATS FOR INDUSTRY. Feuge, R. O. Yearbook of Agr., U. S. Dept. Agr. 1950-51: 529-37.

Many fatty materials intended for industry are modified, or tailored, to give them special properties or characteristics that better adapt them to the process or final products. The more important tailor-made fats are hydrogenated fats, fatty alcohols, sulfated oils, polymerized oils, isomerized oils, and interesterified products. These are grouped, and the chemistry of their preparation is explained, the method of their manufacture is described, and their more important uses are listed.

584. FRACTIONATING FATS FOR NEW PRODUCTS. Feuge, R. O. Yearbook of Agr., U. S. Dept. Agr. 1950-51: 513-19.

The extremely complex chemical nature of naturally occurring fats and oils is explained as a basis for discussing their fractionation into simpler products with properties and uses different from those of the original product. Various techniques for separating fats and fatty acids are briefly described. Discussed in detail are processes for the fractionation of fats and derived products by partial crystallization from solutions. Data are presented to show the effectiveness of the fractionation of naturally occurring oils, of mixtures of fatty acids, and of both natural fats (mixtures of triglycerides) and modified glycerides.

218. MODIFICATION OF VEGETABLE OILS. VI. THE PRACTICAL PREPARATION OF MONO- AND DIGLYCERIDES. Feuge, R. O.; and Bailey, A. E. Oil & Soap 23: 259-64. 1946.

The composition of mixtures of mono-, di-, and triglycerides formed by the reaction of hydrogenated cottonseed oil with glycerol in the presence of an alkaline catalyst was investigated. At temperatures below about 200° C., at equilibrium, and within the range of glycerol concentration in which the reaction product becomes homogeneous, the proportions of free glycerol, mono-, di-, and triglycerides conform closely to a pattern of random distribution with respect to glycerol hydroxyl groups esterified. Tables are given for the calculation of compositions on the basis of random distribution. Products of temperatures above about 200° C. contain minor proportions of some compound or compounds that are high in glycerol content, but are not alpha-monoglycerides. There is evidence that these are probably polymerization products. The composition of reaction products whose combined glycerol content was reduced by high temperature steam distillation under a vacuum (deodorization) was also investigated and does not conform to the random distribution pattern, but shows marked deviations in the direction of increased percentages of diglycerides and decreased percentages of mono- and triglycerides. The composition of deodorized products is independent of the temperature of deodorization or of the presence or absence of the alkaline catalyst. Determinations have been made of the maximum amounts of glycerol miscible in reaction mixtures carried to equilibrium at different temperatures.

65. MEASUREMENT OF THE CONSISTENCY OF PLASTIC VEGETABLE FATS--A STANDARD MICROPENETRATION TECHNIQUE. Feuge, R. O.; and Bailey, A. E. Oil & Soap 21(3): 78-84. 1944.

The theory of plasticity in fats is briefly discussed. The influence of various factors on the consistency of solidified fats has been investigated, a standard technique for making micropenetration tests is proposed, and a quick micropenetration method, applicable as a control in the hydrogenation of fat products, is described. An improved micropenetrometer is described, by means of which it is possible to measure the consistency of fats with a high degree of precision. Micropenetration data are presented on cottonseed, peanut, and soybean oils hydrogenated to different degree, on cottonseed oil blended with various proportions of highly hydrogenated oil, and on various commercial samples of shortening and margarine.

50. MODIFIED HERSHBERG MELTING-POINT APPARATUS. Graff, M. M. Ind. Eng. Chem. 15: 638. 1943.

The Hershberg precision melting-point apparatus has been modified so that melting-point temperatures of low-melting solids may be maintained to within 0.1° C. over a longer period of time. Heating is controlled by a variable-voltage transformer.

48. MODIFICATION OF VEGETABLE OILS. II. A COCOA BUTTER SUBSTITUTE FROM COTTONSEED OIL. Kraemer, E. A.; Smith, B. A.; and Bailey, A. E. Oil & Soap 20(11): 235-40. 1943.

Selective hydrogenation followed by fractional crystallization from a solvent has been used to prepare from cottonseed oil a hard butter very similar to cocoa butter. The new product differs somewhat in composition from cocoa butter, due to an unavoidable content of iso-oleic acid. Examination of the new product by micropenetration technique, by a standard solidification test, and by means of the dilatometer reveals minor physical differences between it and cocoa butter. The new fat also has a slightly longer plastic range than cocoa butter; it supercools less strongly and contracts slightly less upon solidification. Polymorphism is less pronounced. A relatively low yield of the new fat is inherent in the process for preparing it, a yield of approximately 28 percent being obtained in the laboratory. The residual 72 percent is suitable for use as a hardening agent in shortening and similar products.

OIL - NEW PRODUCTS - PATENTS

3356. 4-(2 ALKENOYL, OR 2 BROMO ALKANOYL)-MORPHOLINES. Holmes, R. L.; Moreau, J. P.; and Novak, A. F. U. S. Pat. No. 3,294,794, December 27, 1966.

This invention relates to certain new derivatives of fatty acids, more particularly to the morpholides obtained by reacting morpholine with alpha-beta-unsaturated acids, with 2-bromodecanoic acid, and to propargyl. 2-bromodecanoate. The compounds that are the subject of this invention are characterized by the fact that as growth inhibitors they are effective against a variety of pathogenic molds.

3355. N-OLEOYL, N'-CARBETHOXY PIPERAZINE. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,294,802, December 27, 1966.

This invention relates to N-acyl derivatives of cyclic imines. These materials are useful as plasticizers for vinyl-type resins. The particular compound of this invention is N-oleoyl-N'-carbethoxy piperazine.

3348. MORPHOLIDES OF A MIXTURE OF EPOXIDIZED, SATURATED, AND MONOUNSATURATED FATTY ACIDS. Magne, F. C.; Skau, E. L.; and Mod, R. R. U. S. Pat. No. 3,291,629, December 13, 1966.

This invention relates to the morpholides of mixtures of long-chain saturated and unsaturated fatty acids and to the epoxy derivatives thereof. These mixed morpholides are efficient primary solvent plasticizers for the polymers and copolymers of vinyl chloride.

3346. PROCESS OF CONTROLLING MICROBIAL ACTIVITY. Mod, R. R.; Skau, E. L.; Fore, S. P.; Magne, F. C.; Novak, A. R.; Dupuy, H. P.; Ortego, J. R.; and Fisher, M. J. U. S. Pat. No. 3,285,812, November 15, 1966.

This invention relates to a group of new nitrogen-containing organic compounds. The new compounds are substituted amides and they are characterized by the fact that they are effective as growth inhibitors against a variety of micro-organisms that include pathogenic bacteria, yeasts, and molds.

3345. PROCESS OF CONTROLLING MICROBIAL ACTIVITY. Mod, R. R.; Skau, E. L.; Fore, S. P.; Magne, F. C.; Novak, A. F.; Dupuy, H. P.; Ortego, J. R.; and Fisher, M. J. U. S. Pat. No. 3,285,809, November 15, 1966.

This invention relates to a group of new nitrogen-containing organic compounds. The new compounds are substituted amides and they are characterized by the fact that they are effective as growth inhibitors against a variety of micro-organisms that include pathogenic bacteria, yeasts, and molds.

3336. PROCESS OF PREPARING STABLE TRIGLYCERIDES OF FAT FORMING ACIDS. Bradshaw, B. L.; Feuge, R. O.; and Lovegren, N. V. U. S. Pat. No. 3,270,040, August 30, 1966.

This invention relates to a process for producing stable fatty triglycerides and more particularly to a process for producing the thermodynamically stable polymorphic form of solid fatty triglycerides.

3335. N-OLEOYLPYRROLIDINE. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,270,033, August 30, 1966.

This invention relates to certain compounds that are N-acyl derivatives of cyclic imines, to some unique mixtures of N-acyl derivatives of cyclic imines, and to plastic compositions, the plasticizer component of which is at least one of the compounds or one of the unique mixtures that are the subject of this invention.

3325. VINYL CHLORIDE POLYMERS PLASTICIZED WITH MORPHOLIDES OF FATTY ACIDS AND DIMERIZED FATTY ACIDS. Magne, F. C.; Skau, E. L.; and Mod, R. R. U. S. Pat. No. 3,260,692, July 12, 1966.

This invention relates to unique morpholides. It provides the morpholides of mixtures of long-chain saturated and unsaturated fatty acids and the epoxy and dimer derivatives thereof that can reproducibly be produced from the fatty acids found in natural glycerides. These mixed morpholides are efficient primary solvent plasticizers exhibiting good compatibility with homopolymers and copolymers of vinyl chloride.

3210. FATTY ACID AMIDES AND ESTERS THEREOF. Mod, R. R.; Skau, E. L.; Fore, S. P.; Magne, F. C.; Novak, A. F.; Dupuy, H. P.; Ortego, J. R.; and Fisher, M. J. U. S. Pat. No. 3,250,794, May 10, 1966.

This invention relates to certain new nitrogen-containing organic compounds; to derivatives of aliphatic acid amides that exhibit antimicrobial activity. The substituted amides are characterized by the fact that as growth inhibitors, they are effective against a variety of micro-organisms that includes bacteria, yeasts, and molds, many of which are pathogenic.

3209. VINYL CHLORIDE POLYMERS PLASTICIZED WITH MIXED MORPHOLIDES OF DIMERIZED FATTY ACIDS. Magne, F. C.; Skau, E. L.; and Mod, R. R. U. S. Pat. No. 3,250,635, May 10, 1966.

This invention relates to unique morpholides--morpholides of mixtures of long-chain saturated and unsaturated fatty acids and the epoxy and dimer derivatives thereof that can reproducibly be produced from the fatty acids found in natural glycerides. These mixed morpholides are efficient primary solvent plasticizers exhibiting good compatibility with homopolymers and copolymers of vinyl chloride.

3208. N-ACYL DERIVATIVES OF CYCLIC IMINES. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,248,396, April 26, 1966.

This invention relates to certain compounds that are N-acyl derivatives of cyclic imines, to some unique mixtures of N-acyl derivatives of cyclic imines, and to plastic compositions, the plasticizer component of which is at least one of the compounds or one of the unique mixtures that are the subject of this invention.

3090. DIESTERAMIDE PLASTICIZERS. Magne, F. C.; Mod, R. R.; and Skau, E. L. U. S. Pat. No. 3,226,403, December 28, 1965.

This invention relates to a plastic composition, the plasticizer component of which is a novel class of amides. The amides have the ability to plasticize both the hydrophilic and the hydrophobic type of resins and in addition are characterized by their ability to confer softening characteristics on nitrile rubbers.

3088. N-DECANOYLTETRAHYDROQUINOLINE. Mod, R. R.; Skau, E. L.; Fore, S. P.; and Magne, F. C. U. S. Pat. No. 3,223,708, December 14, 1965.

This invention relates to certain new nitrogen-containing organic compounds. More particularly, this invention relates to derivatives of aliphatic acid amides that exhibit antimicrobial activity.

3087. N-ACYL DERIVATIVES OF CYCLIC IMINES. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,222,203, December 7, 1965.

This invention relates to certain compounds that are N-acyl derivatives of cyclic imines, to some unique mixtures of these derivatives, and to plastic compositions. It relates to N, N-disubstituted long-chain aliphatic amides and to certain other new amide plasticizers wherein the acyl component is derived from dibasic alkanoic acids; epoxidized alkanoic, alkenoic, or alkandioic acids; dimer acids; and cycloalkanoic, cycloalkenoic, cycloalkandioic, or cycloalkendioic acids which contain from 10 to 18 carbon atoms and in which acids the unsaturation, if present, may be in the cyclic group, the side chain, or both, such as the naphthenic, terpene-derived, cyclized linoleic acids and their epoxy derivatives. This invention also relates to certain mixtures of these amides.

3085. MORPHOLIDES OF EPOXIDIZED FATTY ACIDS. Magne, F. C.; Skau, E. L.; and Mod, R. R. U. S. Pat. No. 3,219,664, November 23, 1965.

This invention rel ates to unique mixed morpholides. More particularly, the invention provides the morpholides of mixtures of long-chain saturated and unsaturated fatty acids and the epoxy derivatives thereof that can reproducibly be produced from the fatty acids found in natural glycerides. These mixed morpholides are efficient primary solvent plasticizers exhibiting good compatibility with polymers and copolymers of vinyl chloride.

3084. N-OLEOYL-2-ETHYLETHYLENIMINE. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,219,659, November 23, 1965.

This invention relates to certain compounds that are N-acyl derivatives of cyclic imines, to some unique mixtures of these derivatives, and to plastic compositions. It relates to N, N-disubstituted long-chain aliphatic amides and to certain other new amide plasticizers wherein the acyl component is derived from dibasic alkanoic acids; epoxidized alkanoic, alkenoic, or alkandioic acids; dimer acids; and cycloalkanoic, cycloalkenoic, cycloalkandioic, or cycloalkendioic acids, This invention also relates to certain mixtures of these amides.

3082. VINYL CHLORIDE RESINS PLASTICIZED WITH N-ACYL DERIVATIVES OF SUBSTITUTED PIPERIDINE. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,219,614, November 23, 1965.

This invention relates to certain compounds which are N-acyl derivatives of cyclic imines, to some unique mixtures of these derivatives, and to plastic compositions. It relates to N, N-disubstituted long-chain aliphatic amides and to certain other new amide plasticizers wherein the acyl component is derived from dibasic alkanoic acids; epoxidized alkanoic, alkenoic, or alkandioic acids, dimer acids; cycloalkanoic, cycloalkenoic, cycloalkandioic, or cycloalkendioic acids, which acids contain from 10 to 18 carbon atoms and in which acids the unsaturation, if present, may be in the cyclic group, the side chain, or both, such as the naphthenic, terpene-derived, cyclized linoleic acids, and their epoxy derivatives. This invention also relates to certain mixtures of these amides.

3081. VINYL CHLORIDE ROSINS AND BUTADIENE RUBBERS WITH N-ACYL DERIVATIVES OF CYCLIC IMINES. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 3,219,612, November 23, 1965.

This invention relates to certain compounds that are N-acyl derivatives of cyclic imines, to some unique mixtures of these derivatives, and to plastic compositions. It relates to N, N-disubstituted long-chain aliphatic amides and to certain other new amide plasticizers wherein the acyl component is derived from dibasic alkanoic acids, from epoxidized alkanoic acids, and from dimer acids. It also relates to certain mixtures of these amides.

3079. N-BIS (2-BENZOYLOXYETHYL)-FATTY ACID AMIDE ESTERS. Magne, F. C.; Mod, R. R.; and Skau, E. L. U. S. Pat. No. 3,211,766, October 12, 1965.

This invention relates to a plastic composition, the plasticizer component of which is a novel class of amides. These amides have the ability to plasticize both the hydrophilic and the hydrophobic type of resins and, in addition, are characterized by their ability to confer softening characteristics on nitrile rubbers.

3077. FATTY ACID AMIDE-DIESTERS. Magne, F. C.; Mod, R. R.; and Skau, E. L. U. S. Pat. No. 3,207,769, September 21, 1965.

This invention relates to a plastic composition, the plasticizer component of which is a novel class of amides. The amides that form the plasticizer component of the resinous composition, which is the subject of this invention, have the ability to plasticize both the hydrophilic and the hydrophobic type of resins and, in addition, are characterized by their ability to confer softening charteristics on nitrile rubbers.

2956. DIESTERAMIDE PLASTICIZERS. Magne, F. C.; Mod, R. R.; and Skau, E. L. U. S. Pat. No. 3,179,615, April 20, 1965.

This invention relates to a plastic composition, the plasticizer component of which is a novel class of amides. The amides have the ability to plasticize both the hydrophilic and the hydrophobic type of resins and, in addition, are characterized by their ability to confer softening characteristics on nitrile rubbers.

2955. PROCESS OF PREPARING STABLE TRIGLYCERIDES OF FAT-FORMING ACIDS. Feuge, R. O.; and Landmann, W. U. S. Pat. No. 3,170,799, February 23, 1965.

A process whereby fats in thermodynamically unstable form, whether alone or in a mixture wi with nonfat constituents, are transformed to the thermodynamically stable form. This transformation is brought about by subjecting the fatty material to mechanical working to a degree sufficient to force the polymorphic transformation.

2747. ESTERIFICATION PROCESS. Feuge, R. O.; and Willich, R. K. U. S. Pat. No. 3,119,849, January 28, 1964.

This invention relates to an esterification process whereby monoglycerides and diglycerides can be further esterified without the usual attendant side reactions such as interesterification, which latter reaction is characteristic of conventional processes applied to esterification of fatty acid glycerides.

2456. EPOXIDIZED MONOGLYCERIDE DIACETATES AS PLASTICIZERS FOR POLYMERIZED VINYL CHLORIDE. Ault, W. C.; and Feuge, R. O. U. S. Pat. No. 3,050,481, August 21, 1962.

A plastic composition comprising polymerized vinyl chloride and monoepoxystearyl diaceto glyceride as a plasticizer-stabilizer therefor.

2269. PROCESS FOR THE METHYLATION OF VEGETABLE OIL SOAPSTOCKS. Eaves, P. H.; and Spadaro, J. J. U. S. Pat. No. 3,010,977, November 28, 1961.

A process for the production of methylated vegetable oil soapstock from acidulated soapstocks by reaction with methyl alcohol in presence of Twitchell-type catalysis, with system under slight pressure. Unreacted methanol is flash evaporated.

2058. MONOGLYCERIDE DIACETATES. Feuge, R. O.; and Ault, W. C. U. S. Pat. No. 2,895,966, July 21, 1959.

Epoxidized monoglyceride diacetates of vegetable oils which posses utility as plasticizers for synthetic resins.

2054. PURIFYING AND STABILIZING ACIDS AND AMINES. Skau, E. L.; Mod, R. R.; and Magne, F. C. U. S. Pat. No. 2,951,859, September 6, 1960.

This invention relates to the purification of certain fatty acids containing homologs thereof as impurities. Acids and amines that are inherently unstable can be stored in the form of the acid amine compound.

1794. PROCESS FOR MAKING FATTY ACID DIGLYCERIDE, DIESTERS OF DIBASIC ACIDS. Feuge, R. O.; and Ward, T. L. U. S. Pat. No. 2,874,175, February 17, 1959.

A method for producing glycerides in which two molecules of a fatty acid diglyceride are joined in the form of a diester of a straight-chain aliphatic dicarboxylic acid.

1708. VINYL CHLORIDE POLYMERS PLASTICIZED WITH MORPHOLIDE MIXTURE. Magne, F. C.; Skau, E. L.; and Mod, R. R. U. S. Pat. No. 2,863,845, December 9, 1958.

Vinyl chloride polymers plasticized with the morpholides of long-chain fatty acids. The fatty acid morpholides predominate in unsaturated fatty acids, and the mixed morpholides predominate in unsaturated fatty acids, and the mixed morpholides can be produced readily from the fatty acids found in natural glycerides by the method disclosed.

1704. VINYL HALIDE RESINS PLASTICIZED WITH DIACETO-OLEIN AND PHOSPHORIC ACID ESTERS. Magne, F. C.; Skau, E. L.; and Feuge, R. O. U. S. Pat. No. 2,857,348, October 21, 1958.

Vinyl resins plasticized with diaceto-olein and phosphoric acid esters. These plastic compositions exhibit superior low-temperature brittle point, tensile strength, modulus of elasticity, and reduced combustibility.

1559. PURIFICATION OF LONG-CHAIN FATTY ACIDS. Skau, E. L. U. S. Pat. No. 2,816,903, December 17, 1957.

A method for the purification of long-chain fatty acids. The mixed long-chain fatty acids are treated with acetamide to form a liquid simple-phase solution. Crystallization of the acetamide fatty acid compound permits separation of the pure acids.

1486. OBTAINING PURE PALMITIC ACID FROM VEGETABLE OIL ACIDS. Magne, F. C.; Skau, E. L.; and Mod, R. R. U. S. Pat. No. 2,791,596, May 7, 1957.

A process for obtaining pure saturated long-chain fatty acids from a mixture of acids such as occur in natural glyceride fats or oils, comprising formation of the morpholine salts of the mixed fatty acids and separation of the morpholine salts by crystallization techniques.

1337. DETOXIFIED COTTONSEED OIL FOOTS. Pack, F. C.; and Goldblatt, L. A. U. S. Pat. No. 2,746,864, May 22, 1956.

A commercially feasible process for detoxifying the gossypol present in typical cottonseed oil soapstock and the product of detoxified soapstock with oilseed meal compositions that have improved dusting and pelleting properties as a result of the incorporation of the detoxified soapstock.

1336. GLYCERIDIC MIXTURES EXHIBITING UNIQUE PROPERTIES AND PROCESS FOR THEIR PRODUCTION. Feuge, R. O.; Vicknair, E. J.; and Markley, K. S. U. S. Pat. No. 2,745,749, May 15, 1956.

A process for chemically modifying certain glyceridic mixtures so that the modified mixture exhibits the properties of texture, melting point, flexibility, and the like desired for a particular utilization. The process provides for the chemical modification of glycerides by the controlled acylation of mixtures composed of glycerides of long-chain fatty acids which contain substantial portions of monoglycerides.

OIL - GENERAL CHEMISTRY AND INVESTIGATIONS

3237. MASS SPECTROSCOPY: A TOOL NOW AVAILABLE TO THE FATTY ACID CHEMIST. O'Connor, R. T. J. Am. Oil Chemists' Soc. 43: 338A-40A. 1966

Developments in the application of mass spectroscopy to the chemistry of fatty acids are reviewed in an abstract of introductory remarks presented at a symposium on the subject during a meeting of the American Oil Chemists' Society in October 1965. The review covers highlights of the period from 1898 to the present. The author concludes that the mass spectrometer has become a tool for the solution of practical problems in this field.

2734. RELATIVE REACTIVITY TOWARD HYDROGENATION OF THE OLEOYL GROUP IN THE 2- AND 1,3-POSITIONS OF TRIGLYCERIDES. Tumer, H. V.; Feuge, R. O.; Ward, T. L.; and Cousins, E. R. J. Am. Oil Chemists' Soc. 41: 413-15. 1964.

Olive oil was hydrogenated to an iodine value (I. V.) of about 50 under widely different operating conditions. Three types of catalyst were employed. Each catalyst was used at the lowest possible operating temperature and at 170° C. The hydrogenated samples were subjected to lipase hydrolysis to remove a portion of the acyl groups in the 1,3-positions, and the fractions obtained, as well as the unhydrolyzed samples, were analyzed for fatty acid composition and content of trans monoenes. From these data it was concluded that the position of the oleoyl group in the triglyceride molecule is not a factor in the rate of hydrogenation or isomerization.

2733. APPLICATIONS OF MASS SPECTROSCOPY IN FATTY ACID AND LIPID CHEMISTRY. O'Connor, R. T. J. Am. Oil Chemists' Soc. 41: 4, 14, 15, 20, 22, 25, 55. 1964.

Applications of mass spectroscopy to fatty acid and to lipid chemistry are reviewed. The special difficulties encountered in attempts to apply mass spectroscopy to investigations of long-chain fatty acids, their esters and other derivatives and to triglycerides, which arise from the high molecular weight materials encountered, are described, together with instrumentation recently developed to over come these difficulties. With new-type mass spectrometers, capable of measuring the mass spectra of large molecules, the potential applications of this technique to the identification of long-chain fatty acids, esters and glycerides, the differentiation of various members of homologous series, and the analysis of various derivatives including oxy-, hydroxy-, methoxy-, epoxy-, etc., compounds are described. The mass spectra of unsaturated as well as saturated compounds are considered, and the especially valuable technique for determining the position of a double bond in a long chain means of deuterization with deuterohydrazine is outlined.

2692. RANEY NICKEL CATALYST OF IMPROVED STABILITY AND REACTIVITY IN THE HYDROGENATION OF TRIGLYCERIDES. Tumer, H. V.; Feuge, R. O.; and Cousins, E. R. J. Am. Oil Chemists' Soc. 41: 212-14. 1964.

The degree of activity of Raney nickel catalysts in the hydrogenation of triglycerides has been found to vary considerably with and to depend upon unknown factors in the original alloy. Highly active preparations of Raney nickel rapidly lost their activity when stored in absolute ethanol because of the formation of acetaldehyde. Loss of activity was associated with the amount of residual aluminum in the catalyst. The nickel was found to dissolve to some extent in the acetaldehyde-ethanol solution. Catalysts deactivated by acetaldehyde could be reactivated to a greater degree of activity than that possessed originally be treating with acetic acid. The activity of the original Raney nickel catalyst was preserved to a greater extent by storage in dioxane instead of ethanol.

2676. REPORT OF THE INSTRUMENTAL TECHNIQUES COMMITTEE, AOCS, 1962-1963. O'Connor, R. T., et als. J. Am. Oil Chemists' Soc. 41: 158-60. 1964.

The entire Instrumental Techniques Committee of the American Oil Chemists' Society and each of its Subcommittees met during the 54th Annual Meeting of the Society, April 22-24, 1963. The Color Subcommittee decided to take no action on possible specifications and methods for surface color pending outcome of a poll regarding industry's interest in this project. Similarly, no action or proposed changes in the Society's Method for measurement of color of oils will be undertaken until a decision has been announced by the National Cottonseed Products Association as to whether they will recommend a change in rules to place trading of cottonseed oils on a bleached color, rather than the present refined color basis. The Gas-Chromatography Subcommittee discussed collaborative results obtained from 13 laboratories on four

samples--menhaden, castor, tall, and dehydrated Oil Methyl Esters. The Spectroscopy Subcommittee decided that no useful purpose could be served by further study of methods to permit determination of hydroxyl number by means of exposed spectroscopy. The group agreed to attempt no further collaborative work until a need for a specific method was presented. The Special Task Group for Preparation of Methyl Esters gave tentative results of a collaborative study underway in seven laboratories for the preparation of methyl esters from fatty acids by the methanol : sulfuric acid and the Methanol : BF_3 methods and from triglycerides by the methanol : sulfuric acid procedure.

2530. ACYLATION DE COMPOSES AROMATIQUES PAR LES ESTERS D'ACIDES GRAS. (Acylation of Aromatic Compounds by Esters of Fatty Acids). (PL-480 GRANT). Richert-Mellier, M. T. Oleagineaux 17: 795-97. 1962.

The article concerns the acylation of phenol and of anisole in the presence of boron trifluoride, by means of saturated triglycerides, and by means of methyl esters of the corresponding fatty acids. Tristearin has been treated with phenol and BF_3, following the technique described previously in the case where the acylating agent is palmitic acid. The reaction is a little exothermic and very slow, and vacuum distillation is necessary to isolate the reaction products: The ortho-hydroxy ketone is in the first fraction, then the para-isomer. The yield of purified ketones is from 54 percent of theory; 46. 5 percent of the para-isomer and 7. 5 percent of the ortho-isomer. If one replaces phenol with anisole, the acylation also takes place with a yield of about 50 percent. The long-chain methyl esters behave like the corresponding fatty acids in their reaction with phenol (or anisole) and BF_3. Starting with methyl stearate, we obtained a yield of 73 percent of theory of para- and ortho-hydroxy ketones, purified simply by crystallizations. However, the methyl esters give good yields of hydroxy ketones only if the aliphatic chain is sufficiently long; for example, the yield of para-hydroxy butyrophenone from methyl butyrate treated with phenol and BF_3 does not exceed 20 percent. In the case of the methy esters, the results obtained with the saturated triglycerides treated with benzene and $AlCl_3$ have shown that the yield of alkylphenylketones diminishes with the length of the fatty chain.

2529. HYDROGENATION OF FATS AND OILS. ISOMERIZATION DURING HYDROGENATION. Cousins, E. R. J. Am. Oil Chemists' Soc. 40: 206-10. 1963.

This presentation includes a survey of the development of the presently accepted theory for the formation of isomers based on a half hydrogenation-dehydrogenation mechanism with atomic hydrogen, an appraisal of the relevant work of recent years for its significance when applied to the problem of isomerization, and certain experimental evidence from other fields of heterogeneous catalysis of interest in the hydrogenation of fats and oils. Effects of temperature, pressure, catalyst concentration, and degree of agitation used in commercial plants on the degree of isomerization are discussed. In addition, the conditions necessary for the proper application of the term "selective" to a hydrogenation are examined. An interpretation of recent work to reduce isomerization while retaining selectivity by extending the normal range of the variables and by the use of solvents is covered. Selected works on catalysis from related fields are used to indicate possible modifications of the isomerization mechanism and, perhaps, to reveal fruitful avenues of research.

2492. REPORT OF THE SPECTROSCOPY COMMITTEE, 1961-62. O'Connor, R. T.; and Chipault, J. R. J. Am. Oil Chemists' Soc. 40: 14, 32, 34. 1963.

The Subcommittee of the Spectroscopy Committee of the American Oil Chemists' Society was appointed to study various methods for converting long-chain fatty acids to their methyl esters chiefly for the purposes of spectrophotometric analyses for gas-liquid phase chromatography and to recommend a specific procedure for adoption by the Society. Collaborative data from these studies were reported. These secondary standards, along with the standards for the determination of trans isomers and methyl esters and triglycerides, will continue to be available through the Society.

2484. SOME FACTORS WHICH INFLUENCE THE AMINO ACID AVAILABILITY IN PROTEIN FOODSTUFFS. Altschul, A. M. Feedstuffs 35(7): 67. 1963.

This is a summary given by Dr. Altschul at the 1962 annual meeting of the Animal Nutrition Research Council in Washington, D. C. References are given.

2480. REACTION OF CYCLOPROPENOID FATTY ACID DERIVATIVES WITH HYDROGEN HALIDES. Bailey, A. V.; Magne, F. C.; Boudreaux, G. J.; and Skau, E. L. J. Am. Oil Chemists' Soc. 40: 69-70. 1963.

The treatment of Sterculia foetida oil with concentrated aqueous HCl or with HBr in acetic acid results in the addition of one hydrohalogen molecule per cyclopropenoid moiety. Iodine value and infrared absorption measurements indicate that the mechanism parallels that of the polymerization of sterculic acid and involves the formation of four isomeric monounsaturated monohalo moieties.

2423. FREEZING POINT BEHAVIOR OF THE TERNARY RECIPROCAL SALT PAIR SYSTEM INVOLVING CYCLOHEXYLAMINE AND 2,2'-DIPRIDYLAMINE SALTS OF STEARIC AND PALMITIC ACIDS. Magne, F. C.; Mod, R. R.; and Skau, E. L. J. Chem. Eng. Data 7: 598-600. 1962.

Additional binary and quasi-binary freezing point data have been obtained to establish the complete freezing point diagram for the ternary reciprocal salt pair system involving the cyclohexylamine and 2,2'-dipyridylamine salts of palmitic and stearic acids. These data account for the apparent anomalies previously reported. The diagram includes five crystallization surfaces, one for each of the individual salts and one for an incongruently melting equimolar compound between the 2, 2'-dipyridylamine salts of stearic and palmitic acids. The composition and temperatures have been found for the eight invariant points in the system, including four binary and one ternary eutectics and one binary and two ternary peritectics. The diagram gives complete information on the solid-liquid phase equilibria for any mixture of these salts during freezing or melting. It can be used to predict the order in which the specific compounds will crystallize, over what temperature ranges, and in what yields.

2417. FREEZING POINT BEHAVIOR OF THE TERNARY RECIPROCAL SALT PAIR SYSTEM INVOLVING THE MORPHOLINE AND 2,2'-DIPYRIDYLAMINE SALTS OF STEARIC AND PALMITIC ACIDS. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 39: 444-47. 1962.

Binary and quasi-binary freezing point data have been obtained to establish the freezing point diagram for the ternary reciprocal salt pair system, involving the morpholine and 2, 2'-dipyridylamine salts of stearic and palmitic acids. The diagram shows that there are six crystallization surfaces representing equilibrium with crystals of MS, MP, DS, DP, DS-DP. The compositions and primary freezing points of the six peritectic and five eutectic mixtures have been determined. From the diagram it is possible to predict for any molten mixture of these four salts under equilibrium conditions the temperature at which crystals of each component will appear on cooling, the yields of these crystals and the composition of the liquid phase at various temperatures, and the temperature at which maximum yields of crystals of the specific salts can be obtained.

2380. GENERAL VIEW OF ALKYLARYLKETONES AND THEIR DERIVATIVES. Blaizot, Pierre. (Institute of Research for Oils and Oleaginous Substances, Paris). Oleagineaux 16: 739-41. 1961. (Translation) (PL 480 GRANT).

In 1953, Silverstein and colleagues at Stanford Research Institute acylated benzene, using triglycerides in the presence of aluminum chloride. This acylation reaction has been taken

up in the laboratories of the Institute of Research for Oils and Oleaginous Substances. Saturated glycerides have been transformed into fatty ketones, consisting principally of stearophenone and laurophenone. Later the classic method of acylation by means of acid chlorides was applied for one part on certain substituted derivatives of benzene, chlorobenzene, and anisole, for example, which did not react by the method of Silverstein; for the other part on the bicyclic hydrocarbons such as naphthalene and tetrahydronaphthalene. Thus the ketone Ar-CO-R was obtained possessing in the aliphatic chain alpha to the carbonyl a particularly active methylene group. Starting from the corresponding alpha-bromo compounds, a series of amine or hydroxyl derivatives have been prepared, certain ones presenting characteristic germicidal properties. By reduction of ketone function, alcohols and glycols have been been obtained, whose esters could possess the properties of plasticizers. The ketone hydroxylated in the ring also possesses the properties of a phenolic OH. Analogous products have been prepared in the naphthalene series.

2331. PARTICIPATION OF TOCOPHEROL DERIVATIVES IN LIPOLYSIS. Ory, R. L.; and Altschul, A. M. Biochem. & Biophys. Research Commun. 7: 370-74. 1962.

Extraction of the castor bean acid lipase with N-butanol containing 2,3-dimercaptopropanol or cysteine removed a lipid cofactor which is required for full activity on cottonseed oil glycerides and triolein but not on tributyrin. This cofactor may be replaced in varying degrees by alpha-tocopherol succinate, alpha-tocopherol, and gamma-tocopherol palmitate but not by alpha-tocopherol phosphate or coenzyme Q-10. Soybean lecithin and a synthetic antioxidant, Ionol, were ineffective cofactor replacements, suggesting that the role of the tocopherol derivatives is not simply that of emulsifier or antioxidant but that they may function in the enzyme reactions involving long-chain glycerides.

2281. BINARY FREEZING POINT BEHAVIOR OF SOME N-SUBSTITUTED AMIDES OF PALMITIC, STEARIC, OLEIC, AND LINOLEIC ACIDS. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Chem. Eng. Data 7: 31-32. 1962.

Binary freezing point data have been obtained for the systems N-benzyloleamide-N-benzyllinoleamide, N-cyclohexyloleamide-N-cyclohexyllinoleamide, N-benzylpalmitamide-N-benzylstearamide, and N-stearoylmorpholine-N-palmitoylomorpholine and compared with those for the corresponding systems for the unsubstituted amides. The substitution of the benzyl or the cyclohyxyl group for one of the amido hydrogens results in the formation of a simple eutectic system instead of the continuous series of solid solutions formed in the corresponding oleamide-linoleamide system. On the other hand, the 1 : 1 molecular compound formation observed in the palmitamide-stearamide system is not prevented either when one of the amido hydrogens of these amides is substituted by benzyl or when both amido hydrogens are substituted as in the morpholine derivatives.

2237. RECENT PROGRESS IN THE APPLICATIONS OF INFRARED ABSORPTION SPECTROSCOPY TO LIPID CHEMISTRY. O'Connor, R. T. J. Am. Oil Chemists' Soc. 38: 648-59. 1961.

During the past 5 years many new applications and original techniques of infrared absorption spectroscopy have been introduced to the fatty acid chemist. Applications include qualitative identification, quantitative determinations, and use of infrared spectra for elucidation of molecular structure. The most popular use of infrared spectra in lipid chemistry has been measurement of C-H bending bands about 10 mμ to detect or determine the trans double bond. As an example of the usefulness of infrared spectra in the elucidation of molecular structure, the work reported on the configuration of sterculic acid may be cited. An outstanding recent advance in infrared spectroscopy of importance to lipid chemistry has been the development of

solid-state spectra. Its role in analyses and in structural problems is discussed. Other advances in applications of infrared absorption spectroscopy to specific problems illustrate its widespread application to lipid chemistry.

2130. THE PREPARATION AND POLYMERIZATION OF SOME LONG-CHAIN MONO-, DI-, AND TRIOLEFINS WITH METAL COORDINATION CATALYSTS. Marvel, C. S.; and Rogers, J. Polymer Sci. 49: 335-52. 1961.

Methods of synthesis for 1, cis-9-octadecadiene, 1, trans-9-octadecadiene, 1, cis-9-cis-12-octadecatriene, 1, 9, 12-octadecatriene with 56 percent trans unsaturation, and several necessary intermediates have been described. The polymerization and copolymerization of these dienes and trienes as well as several long-chain mono-olefins and some branched-chain olefins have been described. The octadecadienes yield soluble homopolymers. The octadecatrienes give only crosslinked polymers, and there is evidence of bond migration. The catalyst system, tri-isobutyl-aluminum-titanium tetrachloride, causes bond migration of 1-octadecene.

2092. THE SEPARATION OF GLYCERIDES OF MIXED FATTY ACID CHAIN LENGTH BY GLASS PAPER CHROMATOGRAPHY. Ory, R. L. J. Chromatog. 5: 153-56. 1961.

A chromatographic separation on glass fiber paper of mixed glycerides of acetic, butyric, stearic, palmitic, and oleic acids in solvents consisting of 2 percent and 5 percent ether in iso-octane has been described. In general, glycerides containing fatty acids of increasing chain length tend to move further up the chromatograms than those of shorter chain length.

2047. FREEZING POINT DATA FOR A PORTION OF THE TERNARY SYSTEM: ACETAMIDE-PALMITIC ACID-STEARIC ACID. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Phys. Chem. 64: 1613-16. 1960.

Freezing point data were obtained for stable, metastable, and unstable crystalline phases in binary mixtures of the 1:1 molecular compounds acetamide-palmitic acid (AP) and acetamide-stearic acid (AS) and for a portion of the ternary system: acetamide-palmitic acid-stearic acid. The equimolar mixture of AP and AS exhibited three freezing points representing stable equilibrium with the high-melting modification of acetamide, metastable equilibrium with crystals of AS, and unstable equilibrium with a crystalline phase of unknown composition, respectively. X-ray long spacing measurements of these solidified 1:1 mixtures indicate the presence of the "C" forms of palmitic and stearic acids and the "A" forms of their 1:1 acetamide compounds. The long spacings were essentially the same whether solidification started from stable or from unstable equilibrium, but the X-ray short spacings and the infrared spectra showed slight characteristic differences.

2045. COLOR INDEX FOR COTTONSEED OILS. Pons, W. A., Jr.; Kuck, J. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 37: 671-73. 1960.

The color index determined by the area under the absorption curve in the region of 400 to 500 mμ is preferred over the A. O. C. S. photometric method in research on cottonseed oil color because the color index gives the more accurate measure of the relative chromogen concentration in cottonseed oils. The evidence that the color index method is more reliable includes: (1) a demonstration that the area under the absorption curve may be used in place of absorbence in the Beer-Lambert equation; (2) a panel score for cottonseed oil color intensity agrees with the color index better than it does with the photometric color; and (3) sources of error in the photometric method that do not occur in the color index method, including those contributed by the high emphasis on absorption at 550 and 670 mμ.

2034. PURE ACID-FREE AMIDES OF C_{18} FATTY ACIDS. THEIR PREPARATION AND BINARY FREEZING POINT BEHAVIOR. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Chem. Eng. Data 5: 478-79. 1960.

Complete binary freezing point data have been obtained for the following seven systems: stearamide-elaidamide, stearamide-oleamide, stearamide-linoleamide, stearamide-stearic acid, oleamide-elaidamide, oleamide-linoleamide, and oleamide-oleic acid. The pure acid-free amides were synthesized from the pure acids by an improved method. The freezing point diagrams indicate solid solution formation between stearamide and the unsaturated amides. An incongruently melting 1:1 molecular compound forms between oleamide and elaidamide and between oleamide and oleic acid. Oleamide forms as unbroken series of solid solutions with linoleamide. This freezing-point behavior emphasizes the advantage of using pure acids in the preparation of the pure amides. It can also be concluded that the freezing point cannot be used as a criterion of purity for these unsaturated amides.

1957. POSITIONAL ISOMERS FORMED DURING THE HYDROGENATION OF METHYL OLEATE. Feuge, R. O,; and Cousins, E. R. J. Am. Oil Chemists' Soc. 37: 267-71. 1960.

To obtain information on the influence of the operating variables on the migration of double bonds during the hydrogenation of the oleoyl group, a series of experiments was carried out with purified methyl oleate. With a dry-reduced, electrolytic nickel type of catalyst, hydrogenations were carried out at various catalyst concentrations, temperatures, and rates of hydrogen dispersion. Hydrogenations also were conducted with three other catalysts: the electrolytic nickel after poisoning with sulfur, palladium, and highly active Raney nickel. The reactions usually were stopped at an iodine value of about 50. Of the several variables, temperature was found to have the most marked effect; as the temperature increased from about 90 to 200° C, the proportion of double bonds remaining in the 9-position decreased from about 74 to 17 percent. Increasing the amount of catalyst and decreasing the rate of hydrogen dispersion increased the amount of migration of the double bonds. In some hydrogenated samples, double bonds were found in the 6- through 14-positions. Under comparable conditions the palladium catalyst produced more positional isomers than did the electrolytic nickel catalyst. Sulfur poisoning apparently had no effect on the distribution of the double bonds. With all catalysts except the Raney nickel, the percentages of double bonds found in the 7- and 8-positions generally were approximately equal to those found in the 11- and 10-positions, respectively. Because double bonds hydrogenate more rapidly as their distance from the ester group increases, it appears that more double bonds actually migrated away from than toward the ester group. With the Raney nickel catalyst, which was used at 30° C., end products were produced which contained more 7- and 8- than 10- and 11-isomers. The amount of trans isomers formed was not proportional to either the degree of hydrogenation or the amount of migration of the double bonds.

1874. PREPARATION AND CHARACTERIZATION OF A NUMBER OF AMINE SALTS OF LONG-CHAIN FATTY ACIDS. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 36: 616-19. 1959.

Eighty-eight amine salts of long-chain fatty acids have been prepared, purified by solvent crystallization, and characterized. Forty-five of these were salts of palmitic acid. The rest included salts of capric, lauric, myristic, stearic, oleic, elaidic, and 12-hydroxystearic acids. A variety of aliphatic, aromatic, and heterocyclic amines, including primary, secondary, and tertiary amines, were investigated. The majority of these gave 1:1 acid-amine compounds on solvent recrystallization of an equimolar mixture of acid and amine. Some of the amines gave no crystallizable salt, and with others the pure salt could not be obtained by this procedure. Under the same conditions the symmetrical alkyl, substituted alkyl, and aralkyl secondary amines investigated gave crystallizable compounds containing two molecules of fatty

acid to one of amine. Molecular compounds of 2-aminopyridine with four molecules of saturated fatty acid were formed by recrystallization starting with a 1 to 4 amine-acid mixture. Surface-tension measurements were made for aqueous solutions of a few of the amine salts.

1867. HYDROBORATION OF FATS. I. POSITIONAL ISOMERISM IN THE METHYL OLEATE HYDROBORATION REACTION. Fore, S. P.; and Bickford, W. G. J. Org. Chem. 24: 920-22. 1959.

It has been found that addition of diborane to the ethylenic bond of methyl oleate proceeds smoothly without significant reduction of the carbomethoxy group. Alkaline hydrogen peroxide oxidation of the tris-(carbomethoxyalkyl)-borane resulted in the formation of an equimolar mixture of 9- and 10-hydroxyoctadecanoic acids, establishing that the hydroboration reaction proceeded nonselectively. Little or no isomerization occurred on heat treatment of these substituted trialkyl boranes.

1866. GLASS PAPER CHROMATOGRAPHY OF THE LONG-CHAIN FATTY ACIDS, BROMINATED DERIVATIVES, AND METHYL ESTERS. Ory, R. L.; Bickford, W. G.; and Dieckert, J. W. Anal. Chem. 31: 1447-48. 1959.

Glass paper impregnated with silicic acid has been used to separate the methyl esters of oleic, dibromostearic, tetrabromostearic, and hexabromostearic acids from each other. The same form of glass paper was used to separate the free acids except that no solvent system was found that would separate dibromo- and tetrabromostearic acids. Alumina impregnated glass paper failed to give as good results as the silicic acid paper for the solvent systems studied.

1861. CORRELATION OF SOLUBILITY DATA FOR LONG-CHAIN COMPOUNDS. II. THE ISOPLETH METHOD OF PREDICTING SOLUBILITIES OF MISSING MEMBERS OF HOMOLOGOUS SERIES. Skau, E. L.; and Bailey, A. V. J. Phys. Chem. 63: 2047-54. 1959.

A new graphical method of correlating solubility data for homologous series of compounds and for predicting the solubilities of missing members of the series is described. It supplements the "isotherm method" so that solubility predictions can be extended over a wider range of temperatures and concentrations and applied to additional homologous series. It is based upon the linear relationship $1/T = a(1/n) + b$, derived from the approximate freezing point depression equation and empirical relationships between the number of carbon atoms, and the heats and entropies of fusion of the members of a homologous series. T represents the absolute temperature at which the saturated solutions of the various homologs in a given solvent contain the same mole fraction of solute. The coefficients a and b are constants. The validity of the isopleth method was established by the fact that the $1/T$ versus $1/n$ "isopleth" plots based upon accurate literature data for a number of systems consisted of a family of straight or only slightly curved lines. The isopleth method can be used (1) to smooth out experimental solubility data, (2) to locate discrepancies, and (3) by graphical interpolation or extrapolation to predict the solubilities of missing members of a series. The published solubility data for the normal hydrocarbons containing 8, 12, 16, and 32 carbon atoms were used to predict the solubilities of those containing 10, 14, 18, 20, 22, 24, 26, 28, 30, and 34 carbon atoms at a number of temperatures in nine solvents. Similar predictions were made for the solubilities of the normal alkyl ketones containing 11, 15, 17, and 21 carbon atoms in 13 solvents and for the following compounds in M hexane: the symmetrical normal alkyl ketones containing 27 and 39 carbon atoms; the symmetrical normal secondary amines containing 18, 20, 22, 26, 30, 32, 34, and 38 carbon atoms; the symmetrical normal tertiary amines containing 30, 42, 48 and 60 carbon atoms; the normal fatty acids containing 11, 13, and 19 carbon atoms; and the fatty acid methyl esters containing 11 and 21 carbon atoms. The combined use of the isopleth and isotherm methods of correlation makes it possible to obtain similar complete solubility data

for other solvents and other homologous series on the basis of a minimum of experimental measurements.

1771. POSITIONAL ISOMERS FORMED DURING THE HYDROGENATION OF METHYL LINOLEATE UNDER VARIOUS CONDITIONS. Cousins, E. R.; Guice, W. A.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 36: 24-28. 1959.

Methyl linoleate was hydrogenated to an iodine value of about 80 under various operating conditions. The temperature, rate of hydrogen dispersion, catalyst concentration, and type of catalyst were varied. The several hydrogenation products were analyzed for content of trans isomers and the positions of the residual double bonds. The pattern of distribution of the double bonds was similar in most of the hydrogenations. The greatest concentration of double bonds was found in the 10-position, and the concentrations in the other positions decreased as the distance from the 10-position increased. The distribution curve tended to be symmetrical, that is, approximately the same number of double bonds was found on each side of the 10-position. Operating conditions that normally tend to increase selectivity and the formation of trans isomers also tended to increase the concentration of double bonds at the 10-position. Greatly lowering the temperature of hydrogenation produced a different pattern of distribution of the double bonds. With nickel catalyst at 110° C. the greatest concentrations of residual double bonds were found at the 9- and 12-positions. More than 50 percent of the total double bonds were in these positions. The several hydrogenation catalysts (nickel, sulfur-poisoned nickel, platinum, and palladium) produced the same pattern of distribution of double bonds when used under identical conditions. Also, the platinum and palladium produced much higher proportions of trans isomers than did the nickel.

1767. CATALYTIC HYDROGENATION OF 9, 10-EPOXYOCTADECANOL AND 9, 10-EPOXYOCTADECYL ACETATE. Fore, S. P.; and Bickford, W. G. J. Org. Chem. 24: 620-22. 1959.

Cis-9,10-epoxyoctadecanol and cis-9-10-epoxyoctadecyl acetate have been hydrogenated in ethanolic solution employing palladium-carbon catalyst. Examination of the reaction products established the fact that nearly equal proportions of the 9- and 10-hydroxy isomers were formed in both cases. These results are in marked contrast to the preferential formation of methyl 10-hydroxyoctadecanoate encountered previously during the catalytic reduction of methyl 9, 10-epoxyoctadecanoate. The difference in results obtained with the two esters is attributed to the relative position of the oxirane center with respect to the acyl and alkoxyl oxygen atoms of the ester.

1765. BINARY FREEZING-POINT DIAGRAMS FOR PALMITIC ACID WITH VARIOUS AMINES AND WITH 2-6 DIMETHYL-GAMMA-PYRONE. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 36: 102-03. 1959.

In connection with the search for molecular compounds which might prove applicable in the purification of long-chain fatty acids, freezing-point data have been obtained for binary systems between palmitic acid and the following: ortho-phenylene-diamine, alpha-picoline, 4-amino--2, 6-dimethylpyrimidine, 2-6-dimethyl-gamma-pyrone, 2-aminothiazole, benzylamine, morpholine, 2-aminopyridine, and 4-aminopyridine. The freezing-point diagrams for the first four of these binary systems are of the simple eutectic type. All the others indicate the existence of at least one crystalline molecular compound containing one or more moles of palmitic acid to one of amine. 2-Aminothiazole forms an equimolar compound. Both benzylamine and morpholine form an equimolar and an incongruently melting 2:1 compound. The position of the amino group in the pyridine ring has a marked effect on the behavior of the aminopyridines. Unlike 2-aminopyridine, which forms a 1:1 and a 4:1 compound with palmitic acid, 3-aminopyridine forms only an equimolar compound, and 4-aminopyridine forms only 4:1 and 2:1 compounds. The latter exists in two polymorphic modifications.

1680. FREEZING-POINT BEHAVIOR OF THE TERNARY RECIPROCAL SALT PAIR SYSTEM INVOLVING THE CYCLOHEXYLAMINE AND MORPHOLINE SALTS OF STEARIC AND PALMITIC ACIDS. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 35(12): 688-91. 1958.

Binary and quasi-binary freezing-point data have been obtained to establish the freezing-point diagram for the ternary reciprocal salt pair system involving cyclohexylamine stearate, cyclohexylamine palmitate, morpholine stearate, and morpholine palmitate. The compositions and freezing points of the ternary peritectic and eutectic mixtures have been determined. Cyclohexylamine palmitate and morpholine stearate are the "stable salt pair." From the diagram it is possible to predict the freezing behavior of any molten mixture of these salts, including the temperature at which crystals of each component will appear on cooling, the yields of these crystals and the composition of the liquid phase at various temperatures, and the temperature at which maximum yields of crystals of the specific salts can be obtained. The data supply fundamental information in connection with the crystallization of mixed amine salts in the purification of palmitic and stearic acids.

1624. THE REACTION OF MERCAPTOACETIC ACID WITH METHYL LINOLEATE AND LINOLEIC ACID. Fore, S. P.; O'Connor, R. T.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 35: 225-30. 1958.

The reaction of mercaptoacetic acid with methyl linoleate and with linoleic acid was investigated. The reaction proceeded at low and erratic rates, with and without catalysts, such as peroxides at various temperatures, but could be accelerated by use of a large excess of mercaptoacetic acid. Addition of 1 mole of mercaptoacetic acid to 1 mole of methyl linoleate resulted in a product containing about 40 percent of mono-adduct. Ozonolysis of the purified mono-adduct yielded approximately equimolar quantities of caproic and azelaic acids, indicating that addition occurred about equally at the 9, 10- and 12, 13-ethylenic bonds. The dicarboxylic acid and the dimethyl ester of the mono-adduct and the tricarboxylic acid and trimethyl ester of the di-adduct of linoleic acid and mercaptoacetic acid were prepared, and the infrared spectra and some physical and chemical characteristics of these products were determined. The infrared spectra of the reaction products were obtained and correlated with functional groups that give rise to them. Bands at about 7.8 and 8.8 mμ, commonly observed in long-chain acids and esters and ascribed to C-O vibrations, are intensified in the sulfur-containing reaction products, suggesting characteristic absorption of C-S compounds at almost identical wavelengths. The formation of adducts was accompanied by a high degree of isomerization of the unreacted ethylenic bonds from the cis to the trans form both in the mono-adduct and in unreacted methyl linoleate. The methyl linoleate recovered contained about 12 percent diene conjugation, but catalytic quantities of mercaptoacetic acid were not effective in inducing conjugation.

1545. X-RAY DIFFRACTION OF MOLECULAR COMPOUNDS OF LONG-CHAIN SATURATED FATTY ACIDS. II. SOME FURTHER OBSERVATIONS ON THE DIFFRACTION OF MOLECULAR COMPOUNDS OF ACETAMIDE AND LONG-CHAIN SATURATED FATTY ACIDS. O'Connor, R. T.; Mod, R. R.; Murray, M. D.; Magne, F. C.; and Skau, E. L. J. Am. Chem. Soc. 79: 5129-32. 1957.

Further examination of the X-ray diffraction patterns of molecular compounds of acetamide with myristic, palmitic, and stearic acids has revealed the existence of two polymorphic forms. Three sets of long spacings have been identified: (1) a weak long spacing identical to that of the "C" form of the parent acid; (2) a shorter long spacing corresponding to a modification equal in length to one molecule of the "A" form of the saturated fatty acid and one molecule of acetamide; and (3) a long spacing corresponding in length to two molecules of the "C" form of the saturated fatty acid and two molecules of acetamide. Each of the polymorphic

modifications of these three acetamide compounds has been shown to form an isomorphous series. The lauric acid addition compound is anomalous, exhibiting (other than the weak spacings identified as identical to those of the parent acid) only one set of the spacings. This single set of spacings differs from the longer-chain homologs as it corresponds to one molecule of the "C" form of lauric acid and one molecule of acetamide and exhibits a considerably different pattern of short spacings. Long and short spacings obtained by the usual powder technique are given in a tabulation. These data permit the identification of any of these addition compounds and of their polymorphic modifications.

1471. CHROMATOGRAPHY OF THE PHOSPHOLIPIDS AND RELATED COMPOUNDS OF GLASS PAPER IMPREGNATED WITH SILICIC ACID. Brown, Mona; Yeadon, D. A.; Goldblatt, L. A.; and Dieckert, J. W. Anal. Chem. 29: 30-31. 1957.

A rapid and convenient chromatographic procedure for the separation of phospholipids and some of their hydrolytic cleavage products is described. Glass fiber paper impregnated with silicic acid is used as the chromatographic medium, and a solvent system composed of phenol, ethyl ether, acetone, and water is the developing solution. Choline-containing compounds are detected on the chromatogram with a phosphomolybdic acid-stannous chloride reagent or with a modified Dragendorff reagent. Compounds containing primary amino groups are located with a ninhydrin reagent. Substances that char are detected also by spraying the chromatogram with concentrated sulfuric acid and heating over a hot plate.

1454. THE MIGRATION OF DOUBLE BONDS DURING THE ISOMERIZATION OF METHYL LINOLEATE WITH PALLADIUM ON CARBON CATALYST. Takemura, K. H.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 34: 15-17. 1957.

The isomerization of methyl linoleate by heating with palladium on carbon catalyst gave an initial rapid rise in the degree of conjugation, which reached a maximum and then gradually decreased. The maximum conjugation obtained was about 24 percent after 4 hours' heating at 200 ± 5° C. with 25 percent by weight of catalyst. Ultraviolet absorption spectra did not indicate the presence of any cyclized aromatic components even after 46 hours' heating at 200° C. but showed the formation of aromatics at 250° C. The limit of conjugation obtained is a result of random migration of the olefinic bonds, rather than dimerization or polymerization reactions.

1447. PURIFICATION OF LONG-CHAIN SATURATED FATTY ACIDS BY RECRYSTALLIZATION OF THEIR MOLECULAR COMPOUNDS WITH ACETAMIDE. Magne, F. C.; Mod, R. R.; and Skau, E. L. J. Am. Oil Chemists' Soc. 34: 127-29. 1957.

A method is described for the purification of lauric, myristic, palmitic, and stearic acid, based upon the recrystallization of the fatty acid-acetamide molecular compounds and subsequent regeneration of the acid by extraction of the acetamide with water. The choice of the crystallization solvent is rather critical. For the purification of lauric and myristic acids, acetone or a one-to-one benzene-acetone mixture by volume proved to be the best solvents; and for palmitic and stearic acids either benzene or a two-to-one mixture by volume of benzene and acetone gave the best results. Since the molecular compounds must be crystallized from concentrated solutions, the separation of the crystals from the mother liquor must be made by centrifugation.

1388. BINARY FREEZING-POINT DIAGRAMS FOR PALMITIC ACID WITH A SERIES OF SUBSTITUTED 2-AMINOPYRIDINES. Mod, R. R.; Magne, F. C.; and Skau, E. L. J. Phys. Chem. 60: 1651-54.

Binary freezing-point data have been obtained for palmitic acid with each of the following 2-aminopyridine derivatives: 2-amino-3-methylpyridine, 2-amino-4-methylpyridine,

2-amino-5-methylpyridine, 2-amino-6-methylpyridine, 2-amino-4, 6-dimethylpyridine, and 2, 2'-dipyridylamine. The binary freezing-point diagrams show that each of these amines forms an equimolecular compound with palmitic acid and, in addition, the 2-amino-3-methylpyrdine forms a 2:1 (2 acid:-1 amine) compound. The freezing points of these molecular compounds and a number of metastable polymorphic modifications have been determined.

1387. BINARY FREEZING-POINT DIAGRAMS FOR 2-AMINOPYRIDINE WITH SATURATED AND UNSATURATED LONG-CHAIN FATTY ACIDS. Mod, R. R.; and Skau, E. L. J. Phys. Chem. 60: 963-65. 1956.

Binary freezing-point data have been obtained for 2-aminopyridine with lauric, myristic, palmitic, stearic, oleic, elaidic, and alpha- and beta-eleostearic acids. The binary diagrams prove the existence of two congruently melting, crystalline, molecular compounds in each case. For the saturated acid systems, the compounds had the compositions RCOOH. - $NC_5H_4NH_2$, and 4RCOOH. $NC_5H_4NH_2$, and for the unsaturated acid systems, R'COOH. $NC_5H_4NH_2$ and 2R'COOH. $NC_5H_4NH_2$. If the deviation from ideal freezing-point depression can be attributed to molecular compound formation alone, the data show that the degree of dissociation of the acid-amine compound in the molten state varies with the chain length and the degree of unsaturation. For the saturated acids dissociation is greater the longer the chain length of the acid. For the C_{18} acids the dissociation is greater the less the degree of unsaturation.

1311. APPLICATION OF INFRARED SPECTROPHOTOMETRY TO FATTY ACID DERIVATIVES. O'Connor, R. T. J. Am. Oil Chemists' Soc. 33: 1-15. 1956.

History and application of infrared spectroscopy as a tool for qualitative identification, quantitative determinations, and the elucidation of chemical structures of fatty acid materials are reviewed. One of the tables lists 100 absorption bands employed in the application of infrared spectroscopy to fatty acid chemistry. Literature is grouped according to field of application, and 127 references are included.

1239. X-RAY SPECTROSCOPY. Singleton, W. S. J. Am. Oil Chemists' Soc. 32: 612-15. 1955.

Spectroscopy is a means of determining certain properties of materials by utilizing the effects produced by applied energy of various wavelengths. One division of spectroscopy is that in which X-rays are employed as the source of energy, and X-ray spectroscopy can be subdivided into X-ray absorption, fluorescence, and diffraction. In the field of fats and oils, X-ray diffraction is the application most used. By means of diffraction it is possible to determine crystalline structure and the molecular dimensions of crystalline substances. The major concern of this paper is that phase of crystal structure having to do with polymorphism. The paper reports the development, application, and interpretation of X-ray diffraction patterns as an analytical method of determining polymorphism and changes in polymorphic phases in fats, fatty acids, and derivatives. Equipment, methods, and interpretations of results are discussed.

1238. ULTRAVIOLET ABSORPTION SPECTROSCOPY. O'Connor, R. T. J. Am. Oil Chemists' Soc. 32: 616-24. 1955.

An elementary, theoretical discussion of ultraviolet absorption, with particular emphasis on the type of problems its particular characteristics enable it to solve, has been presented. The experimental measurements and methods, both for qualitative detection and for quantitative determinations, are discussed. The simple analysis of alpha-eleostearic acid in fresh tung oil is described as an example of a single-component determination. The alpha- and

beta-eleostearic acid contents of partly isomerized tung oils are used to illustrate the technique and calculations of a multicomponent analysis. An extension of this multicomponent technique, where the measured ultraviolet absorption must be chemically produced, is described and illustrated by the popular simultaneous determination of polyunsaturated fatty acids in vegetable and drying oils. Applications of ultraviolet absorption methods to studies of (1) hydrogenation, (2) autoxidation, (3) vitamin content, (4) drying mechanisms, (5) refining, bleaching, and deodorization processes, and (6) detection and determination of unusual constituents have been described to illustrate the wide scope of applications of this technique in the chemistry of fats and oils.

1237. INFRARED ABSORPTION SPECTRA. O'Connor, R. T. J. Am. Oil Chemists' Soc. 32: 624-33. 1955.

Infrared absorption spectrophotometry is one of the 15 major branches of analytical spectroscopy. Absorption of infrared radiation to produce vibrational-rotational band spectra is discussed theoretically, and the manner in which these spectra are used for qualitative detection and quantitative determination of organic molecular moieties by group frequency correlations is described. The outstanding application of infrared absorption spectroscopy to fat and oil chemistry has been the detection and determination of cis and trans fatty acids, esters, and glycerides. The methods are described, with several illustrations and applications of the technique to hydrogenation and autoxidation studies. Applications of infrared absorption spectroscopy to autoxidation, rancidity, and drying oil properties are described and miscellaneous applications in investigations of glycerides, lipids, and phospholipids cited. The important contributions of infrared spectroscopy to investigations of polymerization and copolymerization reactions and to the elucidation of the molecular structure of fat and oil compounds are mentioned with several illustrations. Miscellaneous uses of infrared absorption spectroscopy by the fatty acid chemist are described, including use of the oxirane ring bands; methods for quantitative determination of hydroxyl, ketone, and ester groups, the position of double bonds in fatty acid type compounds, and the use of infrared spectra to determine chain length. The interesting potential uses of the "fingerprint" techniques to augment these "group frequency" types of investigations are described.

1235. COMPREHENSIVE SPECIFIC GRAVITY TABLES FOR MIXTURES OF GLYCERIDIC OILS WITH COMMERCIAL HEXANE. Skau, E. L.; Magne, F. C.; and Mod, R. R. U. S. Dept. Agr., ARS-72-2. 1955. (Processed.)

Comprehensive composition-temperature-specific gravity data for binary mixtures of glyceridic oil with commercial hexane have been calculated and tabulated. From the tables it is possible to determine the specific gravities of mixtures of any oil of vegetable, animal, or marine origin with commercial hexane at any temperature from 40° to 110° F.; to determine the composition of any such mixture, that is, the percentages of oil and solvent, from its specific gravity; or to determine the rate of change in specific gravity with change in temperature of any such mixture of known composition. It is necessary to know only the specific gravities of the oil and the hexane in question. Derivation of the equation by which the data are calculated is given, together with instructions to enable the user to prepare similar tables for any other solvents.

1173. X-RAY DIFFRACTION AND INFRARED SPECTRA OF MOLECULAR COMPOUNDS OF ACETAMIDE AND LONG-CHAIN SATURATED FATTY ACIDS. O'Connor, R. T.; Mod, R. R.; Murray, M. D.; and Skau, E. L. J. Am. Chem. Soc. 77: 892-95. 1955.

Infrared spectra and X-ray diffraction data have been obtained for the four molecular compounds formed between acetamide and lauric, myristic, palmitic, and stearic acids. The infrared spectra in the rock salt region resemble those of the constituent molecules and are identical for all members of the homologous series. Infrared spectra are not useful for the

identification of the individual molecular compounds. The "long spacing" and "short spacing" have been measured from X-ray diffraction patterns. The "long spacings" of the molecular compounds have been shown to correspond in length to two molecules of saturated fatty acid and two molecules of acetamide. Each pattern also exhibits a weak "long spacing" identical to that of the "C" form of the parent acid.

1170. SOLUBILITY OF 1-MONOSTEARIN IN VARIOUS SOLVENTS. Ward, T. L.; and Singleton, W. S. J. Am. Oil Chem. Soc. 32: 172-75. 1955.

The practical limits of the solubility of pure monostearin in various solvents at different temperatures have been determined for isopropyl alcohol, ethanol, acetone, methanol, and commercial hexane. The synthetic method was employed, in which the temperature of known quantities of solvent and solute was decreased until crystallization of the solute began. This temperature, corrected for supercooling and heat loss to the surrounding bath, was taken as the equilibrium temperature between the known weight of solute and the known weight of solvent. A comparison of the solubility of monostearin in the various solvents at comparative temperatures indicates that its solubility is greatest in isopropyl alcohol and decreases in the order ethanol, acetone, methanol, and hexane.

1169. INFRARED SPECTRA OF MONO-, DI-, AND TRIGLYCERIDES. O'Connor, R. T.; DuPre', E. F.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 32: 88-93. 1955.

The infrared spectra from 2 to 12 microns of chloroform solutions of several glycerides have been measured and studied with a view to possible analytical applications. These spectra are presented, and conclusions that can be obtained from their examination are reported in this paper. The principal bands, common to the spectra of all glycerides, have been tabulated with correlations of the vibrating groups that give rise to them. The analytical significance of the infrared spectra is considered in detail for three specific regions: (1) the O---H stretching vibration region, about 3.0 microns; (2) the C---O stretching vibration region, about 9.0 microns; and (3) the 10.0 micron region of C---H bending, about the C---C group. Analyses of these regions of the spectra indicate that infrared absorption can be used to detect and to determine mono-, di-, and triglycerides in admixtures and that the method for the determination of transisomers can, with modifications, be extended to include analysis of triglycerides.

1084. SOLUBILITY CHARTS FOR HOMOLOGOUS LONG-CHAIN ORGANIC COMPOUNDS. A COMPREHENSIVE GRAPHICAL CORRELATION OF LITERATURE DATA FOR 138 SYSTEMS INVOLVING 11 HOMOLOGOUS SERIES AND 17 SOLVENTS. Boucher, R. E.; and Skau, E. L. U. S. Dept. Agr. ARS-72-1: 7 pp., 138 charts. 1954.

The charts here presented constitute a comprehensive critical review of the published solubility data for 138 systems involving 11 homologous series and 17 different solvents. Such plots have applications in connection with data on the solubilities of members of homologous series. They can be used to smooth out the experimental solubility data, to locate discrepancies caused by experimental error or by an impurity in one sample in the series, and to predict by graphical interpolation or extrapolation the solubility of missing members of the series.

1083. SURFACE AND INTERFACIAL TENSIONS OF SYNTHETIC GLYCERIDES OF KNOWN COMPOSITION AND CONFIGURATION. Benerito, R. R.; Singleton, W. S.; and Feuge, R. O. J. Phys. Chem. 58: 831-34. 1954.

To understand better the fundamental theory and practical applications of fat emulsions, a precise knowledge of the surface tension at various temperatures and the interfacial tensions against water of pure synthetic glycerides was desired. The semimicro method of Ferguson was modified slightly. From the surface tension-temperature data, enthalpy and entropy

changes in the surface formation were calculated, and equations developed by the least squares method. For the simple triglycerides containing an even number of carbon atoms, the variation of enthalpy and entropy changes with the number of carbon atoms per acid radical is shown graphically. Synthetic glycerides of known configuration were selected to show effects of degree of unsaturation, degree of esterification, and cis-trans isomerism on the surface phenomena.

1012. AN INTERPOLATIVE METHOD OF CALCULATING SOLUBILITIES OF MISSING MEMBERS OF HOMOLOGOUS SERIES. Skau, E. L.; and Boucher, R. E. J. Phys. Chem. 58: 460-68. 1954.

An equation relating the solubilities of homologous long-chain organic compounds to the number of carbon atoms in the molecule has been developed. It provides a method for determining solubilities for missing members of the series by a graphical correlation of published solubility data; and to locate discrepancies caused by experimental error or by an impurity in one sample in the series. A linear equation, $\log N = a + bn$, is derived from the approximate freezing point lowering equation and the known empirical relationships between the number of carbon atoms, n, and the heats and entropies of fusion of the members of the homologous series. N is the solubility of the homologs (expressed in mole fraction) in a given solvent at a given temperature, and a and b are constants, depending upon the temperature and the system involved. Log N versus n isotherms have been plotted for 125 systems involving ten homologous series in 16 different solvents using published data, and results have been tabulated.

963. THE PHYSICAL PROPERTIES OF FATTY ACIDS, GLYCERIDES, AND DERIVATIVES. THE TECHNICAL LITERATURE AND PATENTS OF THE SOUTHERN REGIONAL RESEARCH LABORATORY. Singleton, W. S. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-361. 1953.

This is an abstract bibliography of 84 publications, published since 1941 by members of the Southern Regional Research Laboratory, giving information on a variety of fatty products--modified fats, mono-, di-, and triglycerides, and oils and fatty acids from cottonseed, peanuts, tung nuts, sesame seed, rice, and various minor oilseeds. Important physical properties studied fall into these categories: expansibility, interfacial tension, plasticity, solubility, spectral and thermal properties, and viscosity and density.

962. THE EFFECT OF POSITIONAL AND GEOMETRICAL ISOMERISM ON THE DILATOMETRIC PROPERTIES OF SOME OCTADECENOIC ACIDS. Singleton, W. S.; and Ward, T. L. J. Am. Oil Chemists' Soc. 30: 592-94. 1953.

Four isomeric octadecenoic acids (oleic, elaidic, petroselinic, and petroselaidic) were prepared and their expansibility was determined. The melting dilation of each acid was calculated and found to increase in the order oleic, petroselinic, petroselaidic, and elaidic.

947. MODIFICATION OF VEGETABLE OILS. XV. FORMATION OF ISOMERS DURING HYDROGENATION OF METHYL LINOLEATE. Feuge, R. O.; Cousins, E. R.; Fore, S. P.; DuPre', E. F. and O'Connor, R. T. J. Am. Oil Chemists' Soc. 30: 454-60. 1953.

Methyl linoleate was prepared from cottonseed oil and progressively hydrogenated in four experiments using temperatures at 150° and 200° C., catalyst concentrations of 0.05 and 0.25 percent nickel, and a high and low rate of hydrogen dispersion at atmospheric pressure. Cottonseed oil was progressively hydrogenated in one experiment for comparative purposes. The hydrogenated methyl linoleate was analyzed for the content of total linoleate, conjugated linoleate, total oleate (including trans isomers), and nonconjugated trans isomers (as methyl

elaidate). It was found that the amount of diene conjugation increased as the temperature and catalyst concentrations increased and as the degree of hydrogen dispersion decreased. Conjugation induced by heat and the nickel catalyst was not a factor. All conjugation was the direct result of hydrogenation.

945. MODIFICATION OF VEGETABLE OILS. XIII. SOME ADDITIONAL PROPERTIES OF ACETOSTEARIN PRODUCTS. Feuge, R. O.; Vicknair, E. J.; and Lovegren, N. V. J. Am. Oil Chemists' Soc. 30: 283-87. 1953.

Acetostearin products were prepared by interacting various proportions of acetic anhydride with a commercially available, molecularly distilled monostearin (monoester content, 91.5 percent) and with a technical grade monostearin (monoester content, 59.0 percent). After water washing and drying of the products, a number of properties were determined.

916. ALKYLATION WITH LONG-CHAIN p-TOLUENESULFONATES. IV. ALKYLATION OF ALCOHOLS AND AMINES WITH n-OCTADECYL p-TOLUENESULFONATE. Shirley, D. A.; Zietz, J. R., Jr.; and Reedy, W. H. J. Organic Chem. 18: 378-81. 1953.

Alkylation of a series of alcohols by n-octadecyl p-toluenesulfonate allows formation of the corresponding n-octadecyl ethers in yields ranging from 30 to 80 percent. Alkylation of a series of aliphatic and aromatic primary amines gives mixtures of N-n-octadecyl and N, N-di-n-octadecyl derivatives in total yields ranging in general from 30 to 70 percent. (These studies were part of a general study of alkylation reactions of long-chain p-toluenesulfonate esters. Previous work has been done on alkylation of phenols, thiophenols, and mercaptans.)

885. HYDROXYSTEARIC ACIDS. II. INFRARED SPECTRA AND X-RAY DIFFRACTION OF 10- AND 12-HYDROXYSTEARIC ACIDS AND METHYL ESTERS, AND OF 9, 10-EPOXYSTEARIC ACIDS. O'Connor, R. T.; Mack, C. H.; DuPre', E. F.; and Bickford, W. G. J. Org. Chem. 18: 693-701. 1953.

The infrared absorption spectra of 10- and 12-hydroxystearic acids, their methyl esters, and the 9, 10-epoxystearic acids from elaidic and oleic acids were determined. The most prominent bands in these spectra were correlated with molecular groupings most likely responsible for them. The hypothesis previously proposed to explain the formation of 10-hydroxystearic acid, rather than a mixture of the 9- and 10-isomers, upon hydrogenation of 9, 10-epoxystearic acid has been supported by infrared interpretations, and a possible explanation for the gelling tendency of 12-hydroxystearic acid as compared to the nongelling tendencies of 10-hydroxystearic acid and the methyl esters is given. The X-ray diffraction patterns for the two hydroxystearic acids and for their methyl esters were obtained, and the interplanar spacing measured from them are given along with their relative intensities.

818. A STUDY OF THE SPECTROPHOTOMETRIC METHOD FOR POLYUNSATURATED FATTY ACIDS IN COTTONSEED OILS AND A COMPARISON WITH CHEMICAL METHODS. O'Connor, R. T.; Stansbury, M. F.; Damare, H. G.; and Stark, S. M., Jr. J. Am. Oil Chemists' Soc. 29: 461-66. 1952.

The fatty acid composition of 48 cottonseed oils, representing a random selection with respect to variety, station, year of growth, and iodine values from 89.8 to 117.0, was determined by the spectrophotometric method. Equations for calculating linoleic acid from spectrophotometric data were examined, and a simplified equation is recommended for its calculation in cottonseed oils and other vegetable oils containing no linolenic acid. A procedure whereby the linolenic acid content is first calculated from the spectrophotometric data is suggested as a criterion for use of the simplified equation. The percentages of linoleic, oleic, and total

saturated fatty acids were compared with previously reported values obtained by calculations from iodine and thiocyanogen values.

817. BINARY FREEZING-POINT DIAGRAMS FOR ACETAMIDE WITH OLEIC AND ELAIDIC ACIDS. Mod, R. R.; and Skau, E. L. J. Phys. Chem. 56: 1016-17. 1952.

Complete binary freezing-point data have been obtained for the stable and unstable forms of acetamide with elaidic acid and with the stable and unstable forms of oleic acid. The diagrams show that acetamide forms a molecular compound with both elaidic (trans) and oleic (cis) acids. Each of these compounds exhibits two incongruent melting points, the one stable and the other metastable. The diagrams show also that the oleic acid compound tends to dissociate less than the elaidic acid compound.

815. SURFACE AND INTERFACIAL TENSIONS, VISCOSITIES, AND OTHER PHYSICAL PROPERTIES OF SOME n-ALIPHATIC ACIDS AND THEIR METHYL AND ETHYL ESTERS. Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 29: 313-17. 1952.

Surface tension, interfacial tension against water, viscosity, density, and refractive index were determined at 75° C. for the saturated, even-numbered, n-fatty acids from C_2 through C_{18} and for the corresponding methyl and ethyl esters. The typical effect of temperature on the various physical constants was demonstrated for one of the fatty acids, myristic acid.

780. MELTING AND FREEZING TEMPERATURES. Skau, E. L. Encyclopedia of Chem. Technol. (Ed. by R. E. Kirk and D. F. Othmer.) Interscience Encyclopedia, Inc. New York, Vol. 8, 839-57. 1952.

The subject is treated under the following headings: energy and volume changes at the melting point; effect of pressure; ideal freezing-point behavior; cooling and heating curves; the theory and interpretation of binary freezing-point composition diagrams for simple eutectic systems and for systems exhibiting the various types of compound and solid-solution formation; a description and critical discussion of the various experimental methods for determining the freezing points of pure compounds and mixtures; determination of molecular weights by freezing-point depression; the use of freezing-point determinations as a criterion of purity; anamalous melting points caused by polymorphism, dynamic isomerism and lipid crystal formation; the relationship between melting point and structure for elements and for inorganic and organic compounds; and applications of the melting and freezing process.

746. PHYSICAL PROPERTIES OF FATTY ACIDS. II. SOME DILATOMETRIC AND THERMAL PROPERTIES OF PALMITIC ACID. Ward, T. L.; and Singleton, W. S. J. Phys. Chem. 56: 696-98. 1952.

Pure palmitic acid has been examined by dilatometric and calorimetric methods. The properties investigated and the values found were expansion of the acid in the solid and liquid states, 0.000280 and 0.000968 ml./g./°C., respectively; melting dilation, 0.1806 ml./g.; specific volume over the range of temperature from solid to liquid; specific heat; heat of fusion, 51.2 cal./g. (13.12 kcal. per mole); and entropy. Equations were developed for expressing the specific heat (Cp) of palmitic acid at any temperature (°C.): (solid state) $Cp = 0.3831 + 0.0013t$; (liquid state) $Cp + 0.4624 + 0.00175t$.

744. RELATION BETWEEN FATTY ACID COMPOSITION AND IODINE VALUE OF COTTONSEED OIL. Stansbury, M. F.; and Hoffpauir, C. L. J. Am. Oil Chemists' Soc. 29: 53-55. 1952.

The regularity in the increase in linoleic and in the decrease in oleic and saturated acids with increase in iodine value of cottonseed oils has been shown by obtaining the regression equation

for the glyceride of each acid on the iodine value by use of the compositional data on 48 samples of oil ranging from 89.8 to 117.0 in iodine value. These equations offer a ready means of approximating the fatty acid composition of cottonseed oils from their iodine values.

743. DILATOMETRIC INVESTIGATIONS OF FATS. VI. MELTING DILATION AS A FUNCTION OF CHAIN LENGTH IN FATTY ACIDS AND THEIR GLYCERYL ESTERS. Singleton, W. S.; and Gros, A. T. J. Am. Oil Chemists' Soc. 29: 149-51. 1952.

Melting dilations have been determined for the even-numbered, saturated n-fatty acids in the series lauric through stearic, and some of their glyceryl esters. These data have been correlated with data obtained previously to determine the relationship between melting dilation and the effective chain length of these compounds. The melting dilations of the mono-, di-, and triglyceryl esters of a fatty acid were found to be proportional to the acid residue content of each compound. The melting dilation of a monoglyceride was one-half that of the diglyceride and one-third that of the triglyceride of the same fatty acid. The increment of melting dilation of the fatty acids and glyceryl esters increased regularly with each addition of two methylene groups, with the end group components exerting a constant effect on the volume change. Equations were developed for calculating the melting dilation of the n-fatty acids and their glycerides as a function of chain length of the fatty acid radical. The melting dilations of those compounds that are unsaturated, or have fewer than 10 carbon atoms, cannot be calculated by these equations.

740. BINARY FREEZING-POINT DIAGRAMS FOR PALMITIC ACID WITH SUBSTITUTED ACETAMIDES AND OTHER AMIDES. Magne, F. C.; Hughes, E. J.; Mod, R. R.; and Skau, E. L. J. Am. Chem. Soc. 74: 2793-95. 1952.

Complete binary freezing-point data were obtained for palmitic acid with the formamide propionamide, n-butyramide, isobutyramide, chloroacetamide, phenoxyacetamide, phenylacetamide, stearamide, and benzamide. All these systems except that with formamide proved to be of the simple, eutectic type. The system with formamide showed a region of immiscibility in the liquid state. Binary systems of palmitic acid with succinimide, malonamide, and urea showed regions of immiscibility in the liquid state. In none of the systems was there evidence of a molecular compound of palmitic acid with the amide, such as was found with acetamide.

739. MOLECULAR COMPOUND FORMATION BETWEEN ACETAMIDE AND LONG-CHAIN SATURATED FATTY ACIDS. Magne, F. C.; and Skau, E. L. J. Am. Chem. Soc. 74: 2628-30. 1952.

Binary freezing-point diagrams that have been constructed for each of the polymorphic forms of acetamide with lauric, myristic, palmitic, and stearic acids give conclusive evidence of molecular compound formation between acetamide and each of these acids. The general formula for these compounds is $RCOOH.NH_2COCH_3$. Cryoscopic molecular weight determinations show that in dilute 1,4-dioxane solutions acetamide is 40.4 percent associated, myristic acid is not appreciably associated, and the equimolecular compounds of acetamide and the fatty acids are from 93.5 to about 97 percent dissociated, depending upon the chain length of the fatty acid.

661. DILATOMETRIC INVESTIGATIONS OF FATS. V. VOLUME CHANGES ASSOCIATED WITH THE POLYMORPHISM AND MELTING OF MONOSTEARIN. Singleton, W. S.; and Vicknair, E. J. J. Am. Oil Chemists' Soc. 28: 342-43. 1951.

1-Monostearin, 99.2 percent pure, was prepared and the polymorphic forms, known as sub-alpha (a_s), alpha (a), beta prime (B'), and beta (B), were obtained, and their expansibilities were determined over the temperature range from the solid to the liquid state. The

melting dilation of each form was calculated from the observed expansibilities. From the values for the expansibility, density, and melting dilation, the specific volume of any of the polymorphic forms of monostearin at any temperature from the solid to the liquid state may be calculated. The melting dilations of stearic acid, monostearin, and tristearin are in direct proportion to the mole percentage of stearyl groups present in each compound. By means of this relationship a melting dilation for distearin was calculated.

648. MODIFICATION OF VEGETABLE OILS. XI. THE FORMATION OF TRANS ISOMERS DURING THE HYDROGENATION OF METHYL OLEATE AND TRIOLEIN. Feuge, R. O.; Pepper, M. B., Jr.; O'Connor, R. T.; and Field, E. T. J. Am. Oil Chemists' Soc. 28: 420-26. 1951.

During the hydrogenation of methyl oleate, as much as 38 percent of trans isomers formed while the first 10 percent of methyl stearate was formed. The rate of formation of trans isomers was increased by increasing the temperature, increasing the catalyst concentration, and decreasing the degree of dispersion of the hydrogen. The hydrogenation of methyl oleate always resulted in the establishment of an equilibrium between cis and trans isomers, and the concentration of trans isomers was always 67 percent, calculated on the basis of total unsaturated constituents. It is concluded that all of the iso-oleic acids adsorbed hydrogen at the same rate as oleic acid and were adsorbed and desorbed from the nickel catalyst with equal ease. During the hydrogenation of triolein trans isomers are formed at a slightly lower rate than in the hydrogenation of methyl oleate. Partial hydrogenation of triolein also resulted in the establishment of an equilibrium between cis and trans isomers of oleic acid, but at values of less than 67 percent of trans constituents (based on the total unsaturated constituents) observed with methyl oleate.

560. THE INFRARED SPECTRA OF SATURATED FATTY ACIDS WITH AN EVEN NUMBER OF CARBON ATOMS FROM CAPROIC, C_6 (HEXANOIC), TO STEARIC, C_{18} (OCTADECANOIC), AND OF THEIR METHYL AND ETHYL ESTERS. O'Connor, R. T.; Field, E. T.; and Singleton, W. S. J. Am. Oil Chemists' Soc. 28: 154-60. 1951.

Before infrared spectroscopy can be applied to fatty acid and vegetable oil chemistry to any appreciable extent, considerable data with reference curves on appropriate pure compounds will be required. This paper presents the infrared absorption data from 1 to 12μ of the 13 straight-chain, saturated, monobasic fatty acids from caproic C_6 to stearic C_{18} and of their methyl and ethyl esters, all measured in chloroform solution against the chloroform solvent by mill-type readings. The complete curves are presented with the wavelength positions of the maxima of the most prominent bands indicated. Band assignments correlating most of these bands to molecular structure have been made from information obtained from infrared literature. Intensity relationships throughout the homologous series have been computed for several of the bands, and the significance of the relationship obtained is pointed out. The data should be useful to future applications of infrared spectroscopy to vegetable oil research problems.

551. MODIFICATION OF VEGETABLE OILS. X. EFFECT OF MONOGLYCERIDES ON THE INTERFACIAL TENSION OF OIL-WATER SYSTEMS. Gros, A. T.; and Feuge, R. O. J. Am. Oil Chemists' Soc. 28: 1-4. 1951.

Monoglycerides were prepared from oleic and linoleic acids and from each of the even-numbered, saturated acids from C_8 to C_{18} by reaction of the pure acids with glycerol in the presence of an alkaline catalyst. The reaction products were purified to remove soaps, free fatty acids, and free glycerol, and to concentrate the monoglycerides. The interfacial tensions of oil-water systems containing the concentrated monoglycerides were investigated. Observations were made of the effect of the length of the fatty acid chain in lowering the

interfacial tension at a cottonseed oil-water interface at 70° C. using the monoglycerides of the saturated acids from C_8 to C_{18}. The quantities of monoglycerides required to reduce the interfacial tension to various values were determined. Measurements were made at 50°, 70°, and 90° C. to determine the effect of temperature on the lowering of interfacial tension. The effect of unsaturation on the surface activity of monoglycerides was evaluated by comparing the monoglycerides of stearic, oleic, and linoleic acids. Interfacial tension measurements were made using cottonseed oil, mineral oil, and amyl acetate, successively, as the oil phase to establish the effectiveness of glyceryl monostearate as a surface active agent with widely different types of oily materials.

460. PHYSICAL PROPERTIES OF FATTY ACIDS. I. SOME DILATOMETRIC AND THERMAL PROPERTIES OF STEARIC ACID IN TWO POLYMORPHIC FORMS. Singleton, W. S.; Ward, T. L.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 27: 143-46. 1950.

The expansion of stearic acid in both solid and lipid states, its melting dilation, specific heat, heat of fusion, and entropy have been determined. The transition of the transparent, unstable B-form (long diffraction spacing) of stearic acid obtained by crystallization from benzene at room temperature, to the stable C-form (shorter diffraction spacing) occurred rapidly at 52.9° C. and at a slower rate at a considerably lower temperature (35.2°). Crystallization of stearic acid from hot glacial acetic acid yielded the stable modification, or C-form, and the unstable modification, or B-form, at room temperature. The temperature at which crystallization occurred also determined the crystalline form (B- or C-spacing) of stearic acid that was obtained from benzene solution. Crystallization at temperatures above the transition point produced only the C-form of acid, and below the transition point only the B-form of acid, irrespective of solvent used.

450. NOMOGRAPHS FOR CALCULATING THE FATTY ACID COMPOSITION OF OILS AND FATS FROM IODINE AND THIOCYANOGEN VALUES. Hussain, S. A.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 27: 206-10. 1950.

Nomographs have been constructed for use in calculating the glyceride composition of oils and fats from iodine and thiocyanogen values and their application is described. Equations adopted by the American Oil Chemists' Society relating the glyceride composition with the iodine and thiocyanogen values of an oil were used for constructing the nomographs. The glyceride compositions of various oils calculated from the nomographs were in good agreement with those obtained by calculation from the equations.

448. MODIFICATION OF VEGETABLE OILS. IX. PURIFICATION OF TECHNICAL MONOGLYCERIDES. Feuge, R. O.; and Gros, A. T. J. Am. Oil Chemists' Soc. 27(4): 117-22. 1950.

Experiments with technical monoglycerides prepared from cottonseed oil stearin by the conventional procedure were conducted to determine how readily the monoglycerides in the end products can be concentrated by processes adaptable to large-scale production. A number of the experiments were concerned with the removal of free glycerol, but the greatest effort was directed toward fractionation of the glycerides. Methods of fractional crystallization of glycerides from solvents were the most successful. For example, by changing only the type of solvent employed it was possible to change the monoglyceride content of the fatty portion in the filtrate from 10 to 92 percent. In the 92 percent concentration, over 70 percent of the total monoglycerides in the system were found in the filtrate. At somewhat lower monoglyceride concentrations, appreciably greater portions of the monoglyceride fraction were found in the filtrate.

424. PHASE INVESTIGATIONS OF FATS. III. SYSTEMS CONTAINING OLEIC AND PALMITIC ACIDS AND AN ORGANIC SOLVENT. Singleton, W. S. J. Am. Oil Chemists' Soc. 26: 332-36. 1949.

The ternary systems, oleic acid-palmitic acid-commercial hexane and oleic acid-palmitic acid-acetone, containing varying amounts of the three components were equilibrated at 0°, -10°, -20°, -30°, and -40° C. From compositional data of the liquid and solid phases in equilibrium at each isotherm, ternary phase diagrams were constructed. From these diagrams, which are reproduced in the article, it is possible to predict the degree of separation that can be obtained with any given mixture of oleic and palmitic acids, using either acetone or commercial hexane as solvent. The intersolubilizing effect of oleic acid on palmitic acid was found to be greater in commercial hexane than in acetone, although each acid is more soluble in acetone than in commercial hexane.

399. 2-DECEN-1-OL, 2-UNDECEN-1-OL, 2-DECENAL, 2-UNDECENAL AND DERIVATIVES. Swift, C. E.; O'Connor, R. T.; Brown, L. E.; and Dollear, F. G. J. Am. Chem. Soc. 71: 1512. 1949.

Data are given on the melting points and analyses of 3, 5-dinitrobenzoates and p-nitrophenylurethane of 2-decen-1-ol and 2-undecen-1-ol, and on semicarbazones and 2, 4-dinitrophenylhydrazones of 2-decenal and 2-undecenal. The absorption spectra of the aldehydes, semicarbazones, and 2, 4-dinitrophenylhydrazones exhibit single maxima having characteristic extinction coefficients for these types of compounds.

398. THE ALDEHYDES PRODUCED DURING THE AUTOXIDATION OF COTTONSEED OIL. Swift, C. E.; O'Connor, R. T.; Brown, L. E.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 26: 297-300. 1949.

Spectrophotometric evidence was obtained that Δ^{2}- and $\Delta^{2:4}$- unsaturated aldehydes are produced during the autoxidation of cottonseed oil. The unsaturated carbonyls were converted into semicarbazones and 2, 4-dinitrophenylhydrazones whose characteristics indicated that the oxidation of cottonseed oil produces $\Delta^{2:4}$ - decadienal, Δ^{2}-octenal, and hexanal. Although the exact mechanism of their formation has not been established, these aldehydes are believed to result from the decomposition of the isomeric hydroperoxides of linoleic acid, the principal fatty acid constituent of cottonseed oil.

393. MINUTES OF MEETING OF INTERNATIONAL COMMISSION ON FATS AND OILS, PARTS I, II, AND III. A TRANSLATION. Hoffpauir, C. L. J. Am. Oil Chemists' Soc. 26: 61-5, 106-9, and 151-3. 1949.

This translation incorporates the decisions made on the unified international methods at the London meeting of the International Commission on Fats and Oils in July 1947, and also the plans for collaborative work to be done before the Commission's next meeting. Methods under discussion included those for the sampling and analysis for moisture and oil specified for several oil-bearing seeds. They also included those for alkalis and rosin in soaps, those for soluble and insoluble volatile acids and sterols in fats and oils, and those for the determination of thiocyanogen and peroxide numbers in fats and oil.

347. AUTOXIDATION OF FATS. II. PREPARATION AND OXIDATION OF METHYL OLEATE MALEIC ANHYDRIDE ADDUCT. Bickford, W. G.; Fisher, G. S.; Kyame, Lillian; and Swift, C. E. J. Am. Oil Chemists' Soc. 25: 254-57. 1948.

The adduct of maleic anhydride and methyl oleate was prepared and characterized. Its physical and chemical properties indicate that it is composed of one mole each of the

reactants and that the double bond of methyl oleate is not saturated. Oxidation fission showed that the adduct consisted of a mixture of four isomers that yielded octanoic, nonanoic, suberic, and azelaic acids on ozonolysis. Isolation of these four acids from the fission products indicates that the addition reaction probably proceeds by a mechanism analogous to that proposed by other writers for autoxidation. The data appear to support the free radical chain mechanism for the autoxidation of fats.

346. AUTOXIDATION OF FATS. I. PREPARATION AND OXIDATION OF ALKYLBENZENE-MALEIC ANHYDRIDE ADDUCTS. Bickford, W. G.; Fisher, G. S.; Dollear, F. G.; and Swift, C. E. J. Am. Oil Chemists' Soc. 25: 251-54. 1948.

The experiments described show that certain alkylbenzenes having a hydrogen atom on the alpha carbon atom react with maleic anhydride to form compounds analogous to the hydroperoxides produced by autoxidation of the alkybenzenes. The structure of these products was determined and the results applied, together with other data, to demonstrate that the reaction proceeds by a free radical chain mechanism involving the abstraction of a hydrogen atom from the alpha carbon atom. In view of the analogy between the products obtained in this investigation and in autoxidation, it is suggested that autoxidation is propagated in a similar manner.

330. APPLICATION OF THE LINE-WIDTH METHOD TO THE SPECTROCHEMICAL ANALYSIS OF OILS, FATS, AND RELATED SUBSTANCES. O'Connor, R. T.; Heinzelman, D. C.; and Jefferson, M. E. J. Am. Oil Chemists' Soc. 25: 408-14. 1948.

The line-width method applied to the spectrochemical analysis of oils and fats, in conjunction with an improved ashing technique, permitted quantitative determination of copper, iron, manganese, nickel, and tin in quantities as low as 1 part in 10 million. The procedure was critically examined by precision tests on commercial and experimental samples, by accuracy tests on synthetic samples, and by recovery tests. Results of actual analysis of 30 vegetable oils and fats indicate its usefulness as a research tool, especially in trace element analyses of organic materials that are low in ash content.

323. DECOMPOSITION OF METHYL HYDROPEROXIDE OLEATE. Swift, C. E.; Dollear, F. G.; Brown, L. E.; and O'Connor, R. T. J. Am. Oil Chemists' Soc. 25: 39-40. 1948.

One of the decomposition reactions of methyl hydroperoxide oleate is fission to produce alpha-, beta-unsaturated carbonyl compounds, one of which is 2-undecenal. Qualitative evidence of the presence of the decomposition mixture of analogs and homologs of this aldehyde was obtained in the course of the isolation and purification of 2-undecenal. Aldehydes of the type represented by 2-undecenal readily undergo oxidation, reduction, and isomerization, thus making it possible to account for many of the end products that have been qualitatively and quantitatively identified in various types of oxidizing fat systems.

322. PHASE INVESTIGATIONS OF FATS. II. SYSTEMS CONTAINING OLEIC AND STEARIC ACIDS AND AN ORGANIC SOLVENT. Singleton, W. S. J. Am. Oil Chemists' Soc. 25: 15-20. 1948.

Purpose of this investigation was to determine the composition of the solid and liquid phases existing in equilibrium in the ternary systems, oleic acid-stearic acid-acetone, and oleic acid-stearic acid-commercial hexane, at various temperatures and with varying amounts of the three components. Twelve ternary-phase diagrams, constructed from compositional data of the liquid and solid phases in equilibrium at 0° C., -10° C., -20° C., -30° C., and -40° C., make possible prediction of the degree of separation obtainable from any given

mixture of oleic and stearic acids, using either acetone or commercial hexane as solvent. The intersolubilizing effect of oleic acid on stearic acid, greater in commercial hexane than in acetone, and the possible formation of mixed crystals of oleic and stearic acid were noted. Oleic acid of high purity can be obtained as one of the practical applications of these data.

287. HYDRAZIDES OF p-ALIPHATIC ACIDS. Kyame, Lillian; Fisher, G. S.; and Bickford, W. G. J. Am. Oil Chemists' Soc. 24: 332-34. 1947.

The hydrazides of the normal fatty acid series from valeric through lauric as well as those of myristic, palmitic, and stearic acids have been prepared, and their melting points and mixed melting points have been determined. All of the products are crystalline solids at room temperature and have melting points between 62° and 116° C. The melting differential, approximately 10° between the lowest adjacent members decreases as the series is ascended, being only 3 between palmitic and stearic acid. A plot of the melting points of the hydrazides versus number of carbon atoms gave a smooth curve exhibiting no alternation between even and odd members of the series. The hydrazides are readily prepared from the corresponding esters and appear to be satisfactory derivatives for identifying fatty acids of moderate molecular weight.

284. FATTY ACID COMPOSITION OF HYDROGENATED VEGETABLE OILS. Fisher, G. S.; O'Connor, R. T.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 24: 382-86. 1947.

The fatty acid compositions of a number of unhydrogenated and hydrogenated peanut, cottonseed, soybean, and linseed oils have been calculated from the iodine number, thiocyanogen number, ultraviolet absorption after alkali isomerization, and the content of saturated acids determined by a modified Bertram oxidation method. All the methods gave values in good agreement for the various samples of peanut oil. The iodine-thiocyanogen method and the modified Bertram oxidation method gave values in good accord for cottonseed oils; but the spectrophotometric method gave values for linoleic acid too high in the unhydrogenated and slightly hydrogenated cottonseed oils. Isolinoleic acid was not produced during hydrogenation of cottonseed and peanut oils under the conditions used. Its production during the hydrogenation of soybean oil can be accounted for by hydrogenation of the linolenic acid present in that oil.

254. INTERFACIAL TENSION OF OIL-WATER SYSTEMS CONTAINING TECHNICAL MONO- AND DIGLYCERIDES. Feuge, R. O. J. Am. Oil Chemists' Soc. 24: 49-52. 1947.

Technical mono- and diglyceride products have been prepared from cottonseed oil-stearin, and the effectiveness of these products in lowering the interfacial tension at vegetable oil-water interfaces has been investigated. When both monoglycerides and diglycerides are present in the oil phase, the surface activity of the diglycerides is less than 1/100th that of the monoglycerides on a weight-for-weight basis; hence the interfacial tension is substantially a function of the monoglyceride content. The interfacial tensions against water of such oils as peanut, cottonseed, and soybean are practically the same, and are lowered by practically equal amounts for equal additions of a given monoglyceride preparation. A concentration of 1 percent of monoglycerides in the oil phase will lower the interfacial tension at the oil-water interface to approximately one-half that observed when no monoglycerides are present. A concentration of 6 percent of monoglycerides in the oil phase will lower the interfacial tension approximately to zero.

251. THERMAL PROPERTIES OF FATS AND OILS. VI. HEAT CAPACITY, HEATS OF FUSION AND TRANSITION, AND ENTROPY OF TRILAURIN, TRIMYRISTIN, TRIPALMITIN, AND TRISTEARIN. Charbonnet, G. H.; and Singleton, W. S., J. Am. Oil Chemists' Soc. 24: 140-42. 1947.

Heat capacities of the alpha-and beta-forms of trimyristin, tripalmitin, and tristearin, and the beta-form of trilaurin were measured. The heats of fusion of the beta-forms of these four compounds were determined. Those of the alpha-forms of trimyristin, tripalmitin, and tristearin were calculated from heat-content data. Calculations were also made of the heats of transition, alpha-to beta-form. The molal entropy at 298.16° K. was calculated for the beta-form of each compound.

245. THE HYDROLYSIS OF FATTY ACID CHLORIDES. Bauer, S. T.; and Curet, M. C. J. Am. Oil Chemists' Soc. 24: 36-9. 1947.

Chlorides of octanoic, decanoic, lauric, myristic, palmitic, stearic, oleic, elaidic, and linoleic acids were hydrolyzed at 25° C. in water and the amounts of unchanged acid chlorides determined after different periods of reaction. The chlorides of the longer chain fatty acids, palmitic and stearic, reacted at a more rapid rate than the chlorides of the shorter chain fatty acids. Lauryl chloride appears to be more resistant to hydrolysis than either the chlorides of the lower molecular weight octanoic and decanoic acids or the chlorides of the higher molecular weight myristic to stearic acids. The chlorides of the unsaturated acids, oleic, elaidic, and linoleic, are hydrolyzed less rapidly than stearyl chloride. Elaidyl and myristyl chlorides exhibit the same relative rates of hydrolysis during the first 2 hours of reaction, but after that time the myristyl chloride hydrolyzes more rapidly. The addition of either hydrochloric acid or free fatty acids to the reaction mixture had no pronounced effect on the hydrolysis of the acid chlorides.

236. PREPARATION OF ASH AND SPECTROCHEMICAL DETERMINATION OF TRACES OF METALLIC ELEMENTS IN OILS, FATS, AND RELATED SUBSTANCES. O'Connor, R. T.; Heinzelman, D. C.; and Jefferson, M. E. J. Am. Oil Chemists' Soc. 24: 185-89. 1947.

Because even extremely small concentrations of any metallic elements present in fats or oils may affect quality, a special procedure of ashing fat or oil samples for spectrochemical analysis was developed. It is a modification of a magnesium nitrate ashing method and required initial charring and drying in vacuo followed by ashing in a furnace overnight. Traces of certain metals in ash obtained by this method can be detected when present in only a few parts per million. The preparation of the standards needed to obtain the calibration curves is discussed. Using given spectrum lines, germanium meets the requirements for an internal standard for the determination of aluminum, copper, iron, nickel, and manganese; and lead, for tin and zinc. The use of direct current gives the best results. Spectrograms of typical vegetable oils are shown.

226. THE OXIDATION OF METHYL OLEATE. I. THE PREPARATION, PROPERTIES, AND REACTIONS OF METHYL HYDROPEROXIDE OLEATE. Swift, C. E.; Dollear, F. G.; and O'Connor, R. T. Oil & Soap 23: 355-59. 1946.

The oxidation of the methyl ester of oleic acid, one of the principal acid components of the glycerides of cottonseed oil, was investigated. A method was devised for the preparation and isolation fo the first product of oxidation of methyl oleate, methyl hydroperoxide oleate. Information was obtained relative to the structure and the chemical and physical properties of the hydroperoxide. The hydroperoxide was inactive towards alpha-tocopherol when the two compounds were mixed and stored at room temperature.

198. SPECTRAL PROPERTIES OF LINOLEIC AND LINOLENIC ACIDS PREPARED BY DEBROMINATION. O'Connor, R. T.; Heinzelman, D. C.; Caravella, Maizie; and Bauer, S. T. Oil & Soap 23 : 5-9. 1946.

Linoleic and linolenic acids were separated and purified by modifications of generally accepted methods. Absorption spectra were determined for each of the acids obtained. Extinction curves of the four linoleic acids and the two linolenic acids show successively decreasing absorption owing to the removal of or repression of conjugated impurities by progressively modified and improved methods of purification. The absorption spectrum of the product from each method of preparation was measured in the ultraviolet region from 220 to 320 mμ. The curve for linoleic acid prepared from corn oil showed marked absorption in the regions of diene conjugation, 232 mμ, and of triene conjugation, 268 mμ, and also measurable absorption in the tetraene region, 310 mμ. Another curve for the above acid shows the effect of molecular distillation in its purification. Diene conjugated impurities were almost eliminated, and triene and tetraene constituents were greatly reduced. Two other curves show the continual reduction in conjugated components. Two similar curves are given for the ultraviolet absorption of linolenic acid. The data obtained offer an example of the utility of the spectrophotometer in following the progress of the preparation of a pure compound.

193. THE PREPARATION OF FATTY ACID CHLORIDES. Bauer, S. T. Oil & Soap 23 : 1-5. 1946.

Fatty acid chlorides of lauric, myristic, palmitic, stearic, oleic, elaidic, and linoleic acids were prepared with phosphorus tri- and pentachlorides, oxalyl chloride, and thionyl chloride as chlorinating agents. On the basis of yields, ease of handling, and cost of reagent, phosphorus tri- and pentachlorides are superior for the preparation of the higher fatty acid chlorides for use as intermediates in the preparation of pure saturated fatty acid glycerides of known structure. The unsaturated fatty acid chlorides are best prepared from oxalyl chloride. The use of phosgene failed to effect efficient conversion of the higher fatty acids to the corresponding chlorides. The analysis of the fatty acid chlorides by conversion to anilides provides a simple and easy means for determining the extent of conversion of fatty acids to chlorides. With this method it is possible to follow the course of the reaction to determine when fatty acid is completely converted to fatty acid chloride.

174. PHASE INVESTIGATIONS OF FATS. I. APPARATUS AND TECHNIQUES FOR FAT-SOLVENT SYSTEMS AT LOW TEMPERATURES. Bailey, A. E.; Singleton, W. S.; and Feuge, R. O. Oil & Soap 23 : 201-04. 1946.

An apparatus for equilibrating and separating mixtures of fats and volatile solvents at relatively low temperatures is described, together with the technique for its manipulation. This apparatus consists of a constant-temperature bath controlled by on-and-off operation of a circulating pump through an electronic relay circuit, with a special toluene-filled thermoregulator, used in conjunction with an equilibrium-filtration assembly. The latter is designed so that it can be completely immersed in the bath liquid and transmit heat to the material, and so that the entire operation of equilibration and filtration can be accomplished without removing any part of the apparatus from the bath. With proper manipulation, losses of material are small. Two methods of calculating the composition of the solid and liquid phases are illustrated by data obtained in a typical experiment performed on the system stearic acid-oleic acid-commercial hexane.

159. THE NEW CHEMISTRY AND TECHNOLOGY OF OILS AND FATS. Bailey, A. E. Chronica Botanica Co., Waltham, Mass. 9 : 128-35. 1945.

A review of previous research, together with a summary of the major problems involved in future research with regard to the chemistry and technology of oils and fats, is presented. The topics discussed are: the occurrence of fats and oils; utilization of fats; composition and

structure of natural fats; the preservation of edible fats; the fractionation of fatty materials; polymerized oil products; and the surface activity of fat derivatives. The largest present source of oils comprises the seeds of annual plants, such as cotton, flax, peanuts, and soybeans. The important animal fats are butterfat and the body fats of cattle, hogs, and sheep. The largest and most important use of fats and oils is for food, with use for soap ranking second, and paint and other drying-oil products, third.

156. MODIFICATION OF VEGETABLE OILS. III. FRACTIONAL CRYSTALLIZATION OF FATTY ACIDS FROM SOLVENTS--SEPARATION OF THE SOLID AND LIQUID ACIDS OF COTTONSEED OIL. Singleton, W. S.; Lambou, M. G.; and Bailey, A. E. Oil & Soap 22 : 168-74. 1945.

An investigation is reported of low-temperature crystallization from organic solvents as a means of effecting practical separations of the solid and liquid acids of unhydrogenated and hydrogenated cottonseed oils. In the case of the hydrogenated acids, iso-oleic acids are among the solid acids produced during the course of hydrogenation. At any fixed temperature the most efficient separations were obtained in the highly polar solvents, acetone and methyl acetate.

155. DILATOMETRIC INVESTIGATIONS OF FATS. IV. ESTIMATION OF SOLIDS AND LIQUID IN SOME PLASTIC FATS Singleton, W. S.; and Bailey, A. E. Oil & Soap 22 : 295-99. 1945.

Dilatometric curves were determined over the complete melting range for samples of prime steam lard, oleo oil, all-hydrogenated vegetable shortening, and partly hydrogenated cottonseed oil, to oleic acid, without intermediate desorption of linoleic acid. In both selective and nonselective hydrogenation, the hydrogenation of linolenic acid to linoleic acid and to isolinoleic acid appears to proceed at approximately equal rates.

153. THERMAL PROPERTIES OF FATS AND OILS. V. THE HEAT CAPACITY AND HEAT OF FUSION OF HIGHLY HYDROGENATED COTTONSEED OIL. Oliver, G. D.; and Bailey, A. E. Oil & Soap 22 : 39-41. 1945.

The heat content of a quickly chilled and of a slowly chilled and tempered sample of almost completely hydrogenated cottonseed oil was measured over a temperature range within which there was in each case complete transformation of the oil from a solid to a liquid form. Heat capacity data for the liquid oil and for the quickly chilled and the tempered solid oil were calculated. Equations expressing the changes in heat capacity with temperature were derived. Heat capacity data on highly hydrogenated cottonseed oil are correlated with similar data previously obtained on unhydrogenated and on partly hydrogenated cottonseed oil. The heat of fusion calculated for both the quickly chilled and the tempered solid oil is given.

152. SPECTROPHOTOMETRIC ESTIMATION OF SOYBEAN OIL IN ADMIXTURE WITH COTTONSEED AND PEANUT OILS. O'Connor, R. T.; Heinzelman, D. C.; and Dollear, F. G. Oil & Soap 22 : 257-63. 1945.

A spectrophotometric method is described for the determination of soybean oil in admixture with cottonseed oil to permit an accurate determination of linolenic acid as a criterion of the economic value of an oil mixture and as a guide in oil processing. The precision of the method is limited by variation in composition of the cottonseed and soybean oils in the mixtures. The proposed method can be extended to determine soybean oil in admixture with peanut or any other vegetable oil with negligible linolenic acid content.

143. MODIFICATION OF VEGETABLE OILS. IV. REESTERIFICATION OF FATTY ACIDS WITH GLYCEROL. Feuge, R. O.; Kraemer, E. A.; and Bailey, A. E. Oil & Soap 22 : 202-07. 1945.

The esterification of glycerol and peanut oil fatty acids was studied under reduced pressure, with and without the assistance of various metal chlorides and oxides as catalysts. The uncatalyzed reaction is bimolecular in character but proceeds in two successive stages, of which the latter has the lower velocity constant. Zinc and tin chlorides were outstanding in catalytic activity. The reaction is complex and no longer simply bimolecular. The reaction proceeds at a reasonable speed, i. e. the free fatty acid content of the product is reduced to about 3 percent in 6 hours, with 0. 0008 mole of tin chloride per 100 g. of fatty acids as a catalyst at 175° C., or with a similar amount of zinc chloride as a catalyst at 200° C. Equally rapid esterification is obtained without a catalyst only above 250° C. Other metal chlorides are relatively poor catalysts.

139. DILATOMETRIC INVESTIGATIONS OF FATS. III. THE DENSITY, EXPANSIBILITY, AND MELTING DILATION OF SOME SIMPLE TRIGLYCERIDES AND OTHER FATS. Bailey, A. E.; and Singleton, W. S. Oil & Soap 22 : 265-71. 1945.

Information is given on the expansibility of fats in the solid state and on the volume changes accompanying melting and polymorphic transformations. Dilatometric curves between about -38° C. and temperatures representing the liquid state were determined for a number of pure triglycerides and some commercial fats. From data on the liquid samples, determinations were made on density in both the liquid and the solid states, on expansibility of each state with increased temperatures, and on the dilation accompanying melting. Volume changes accompanying the transformation from one polymorphic form to another were determined. Certain relationships are pointed out among the properties discussed.

138. THERMAL PROPERTIES OF FATS AND OILS. IV. SOME OBSERVATIONS ON THE POLYMORPHISM AND X-RAY DIFFRACTION CHARACTERISTICS OF TRISTEARIN AND A HIGHLY HYDROGENATED COTTONSEED OIL. Bailey, A. E.; Jefferson, M. E.; Kreeger, F. B.; and Bauer, S. T. Oil & Soap 22 : 10-13. 1945.

Melting points and X-ray diffraction patterns are determined for cottonseed oil hydrogenated to an iodine value of less than 1 and for highly purified tristearin. The results for the two materials are compared and point to a well-defined crystal structure, hitherto unreported, for both materials in the lower melting forms. The patterns of the two materials differed in the highest melting forms. A new pattern was observed for tristearin at the intermediate melting point. Four polymorphic forms of the hydrogenated cottonseed oil were detected. The X-ray pattern of the lowest melting form of the hydrogenated oil was similar to that of the corresponding form of tristearin. The pattern of the highest melting form of the hydrogenated oil differed from that of either tristearin or beta-palmitodistearin, the major components of the oil. Distinctive patterns for the intermediate forms of the hydrogenated oil could not be obtained, presumably because of the instability of the lower melting forms at room temperature.

121. LYOPHILIZATION APPARATUS. Pomes, A. F.; and Irving, G. W., Jr. Science 101 : 22. 1945.

The diagram accompanying this communication illustrates a simple, rugged, and inexpensive lyophilization apparatus that has been used successfully for moderately large-scale laboratory work. Its use is discussed.

116. MODIFIED THIOCYANOGEN REAGENT AND METHOD. Lambou, M. G.; and Dollear, F. G. Oil & Soap 22 : 226-32. 1945.

A general reagent applicable to a wide range of thiocyanogen values and capable of reacting with practically all types of fats and oils is described. Features of the method include rigorous purification of reagents; replacement of 25 percent of the volume of glacial acetic acid normally used in preparing the thiocyanogen reagent with an equal volume of carbon tetrachloride; use of finely powdered, dry potassium iodide; and increase in the amount of potassium iodide added before titration from 1.0 gram to twice the equivalent weight calculated for the volume of standard reagent used. Complete exclusion of water from all reagents and glassware is essential.

114. DETERMINATION OF GAMMA-TOCOPHEROL IN VEGETABLE OILS. Fisher, G. S. Ind. Eng. Chem., Anal. Ed. 17 : 224-27. 1945.

Details are presented of a method for determining gamma-tocopherol in the presence of alpha-tocopherol by application of the principle of differential spectral separation after nitric acid oxidation in the presence of acetic acid. The use of the method presupposes the absence of beta-tocopherol. The method was applied to 13 samples of vegetable oils and the gamma-tocopherol contents were determined.

94. THERMAL PROPERTIES OF FATS AND OILS. III. CALORIMETRIC ESTIMATION OF SOLID AND LIQUID GLYCERIDES IN COTTONSEED OIL. Bailey, A. E.; and Oliver, G. D. Oil & Soap 21 : 300-2. 1944.

The relative amounts of solid and liquid glycerides in cottonseed oil and a hydrogenated cottonseed oil have been estimated calorimetrically at different temperatures over the melting ranges of the oils. A correlation has been established between the content of solids in hydrogenated cottonseed oil and the consistency of the oil, as measured by micropenetration. From previous micropenetration data on commercial fat products, it is established that such fats are plastic and easily worked and estimates were made of the percentages of solids and liquid in the fats at different temperatures. The consistency of the fats, as measured by micropenetrations, was different for different fats that contained equal percentages of solids. The consistency, therefore, is not determined solely by the amount of solids present, but also by the character of the solid particles.

93. THERMAL PROPERTIES OF FATS AND OILS. II. HEAT CAPACITY AND LATENT HEAT OF COTTONSEED OIL. Oliver, G. D.; Singleton, W. S.; Todd, S. S.; and Bailey, A. E. Oil & Soap 21 : 297-300. 1944.

The heat contents of cottonseed oil, a hydrogenated cottonseed oil, and a mixture of cottonseed oil and petroleum naphtha have been measured over ranges of temperatures within which there is transformation of the glycerides of the oils from a completely solid to a completely liquid form. From the heat content data, determinations have been made of the heats of fusion of the oils and of the specific heats of the oils in both solid and liquid states. Equations have been derived for expressing the heat capacities of the oils and the oil-solvent mixture, with the oil in either the liquid or solid form. Values found for the specific heat of the liquid oils are in general agreement with values found by previous investigators. The values calculated for the heat of fusion of the oils are much lower than values reported by other workers for highly saturated glycerides. However, a low value for mixed glycerides of saturated and unsaturated fatty acids is to be expected from considerations of molecular structure.

92. THERMAL PROPERTIES OF FATS AND OILS. I. APPARATUS AND TECHNIQUES FOR LOW-TEMPERATURE CALORIMETRY. Bailey, A. E.; Todd, S. S.; Singleton, W. S.; and Oliver, G. S. Oil & Soap 21 : 293-97. 1944.

A low-temperature calorimeter suitable for the examination of fatty materials with moderate accuracy is described and its method of use outlined. The applications of calorimetry in the examination of fats and fatty materials are discussed.

91. NOTES ON THE STABILIZATION OF OXIDIZED FATS BY STEAM DEODORIZATION WITH PHOSPHORIC ACID OR COMMERCIAL LECITHIN. Bailey, A. E.; and Feuge, R. O. Oil & Soap 21 : 286-88. 1944.

When used for stabilization of normal fat containing tocopherols, lecithin or phosphoric acid are equally effective, whether added before or after deodorization. However, if the fat is oxidized until nearly rancid, either of these antioxidants is much more effective if added before the fat is deodorized. There is an optimum amount of each to be used; the most effective stabilization is obtained with about 0. 1 percent lecithin or 0. 004 percent phosphoric acid. Oils deodorized with 0. 1 percent lecithin darkened in the deodorizer, but there was no darkening with amounts of lecithin up to 0. 02 percent. The optimum amount of phosphoric acid (0. 004 percent) had no adverse effect on the color. Phosphorus added in the form of either lecithin or phosphoric acid is not removed from the oils to any large degree by steam deodorization.

87. DILATOMETRIC INVESTIGATIONS OF FATS: II. DILATOMETRIC BEHAVIOR OF SOME PLASTIC FATS BETWEEN 0° C. AND THEIR MELTING POINTS. Kraemer, E. A.; and Bailey, A. E. Oil & Soap 21 : 254-56. 1944.

Dilatometric curves between 0° C. and their melting point have been obtained for lard, butterfat, cottonseed oil, peanut oil, and a commercial margarine oil, a commercial all-hydrogenated vegetable shortening, three samples of soybean oil hydrogenated to different degrees, a hard butter fractionally crystallized from hydrogenated peanut oil, a mixture of tristearin and soybean oil, and a synthetic fat containing equal molar proportions of stearic and oleic acids. The dilatometric curves of volume change in the fat against temperature were in every case composed of a series of straight lines separated by sharp breaks, or transition points. The number of linear sections in the dilatometric curves corresponded in a general way with the known degree of complexity in the glycerides of the fats, and varied from two in the case of the relatively simple stearic-oleic glyceride mixture to at least seven in the case of the all-hydrogenated shortening. Since each break in the curve must correspond to the disappearance of a distinct class of solid glycerides or glyceride complexes, the application of a dilatometry to qualitative and quantitative determination of glyceride composition in fats is suggested.

83. DILATOMETRIC INVESTIGATIONS OF FATS: I. APPARATUS AND TECHNIQUES FOR FAT DILATOMETRY. Bailey, A. E.; and Kraemer, E. A. Oil & Soap 21 (9) : 251-53. 1944.

Dilatometry has been used, alone and in conjunction with calorimetric methods, for investigating phase changes in fats and oils over the partial or complete melting range of these materials. Although dilatometry is simple in principle, useful and reliable results can be obtained only by using well-designed apparatus and observing a number of precautions, some of which are by no means obvious. Apparatus and procedures found satisfactory in experimental use are described and illustrated.

77. TOCOPHEROL CONCENTRATES BY THE FRACTIONAL CRYSTALLIZATION OF COTTONSEED OIL FROM SOLVENTS. Singleton, W. S.; and Bailey, A. E. Oil & Soap 21 : 224-26. 1944.

Tocopherol concentrates equivalent in tocopherol content and antioxygenic activity to molecularly distilled concentrates have been obtained from cottonseed oil by hydrogenating the oil only at solids contents between about 15 and 35 percent. Differences in the solids content of as little as 1 percent are sufficient to cause noticeable differences in the consistency of plastic fats. Within the ranges of temperature and iodine value at which hydrogenated cottonseed oil is plastic, it is estimated that each decrease in iodine value of 1 unit causes the solid glycerides content of the oil to increase approximately 1 percent.

52. THE PREPARATION OF PURIFIED METHYL LINOLEATE BY CHROMATOGRAPHY. Swift, C. E.; Rose, W. G.; and Jamieson, G. S. Oil & Soap 20 (12) : 249-50. 1943.

The successful application of chromatography to the separation of the glycerol esters of fatty acids suggests that the separation of the methyl esters may be effected more easily than separation of the acids themselves. Chromatography of the methyl esters of cottonseed oil fatty acids has been used in preparing purified methyl linoleate. Techniques for accomplishing this without use of the Rollett bromination-debromination procedure are described.

49. SECOND SUPPLEMENT TO BIBLIOGRAPHY ON MOLECULAR OR SHORT-PATH DISTILLATION. Todd, S. S. Oil & Soap 20 (10) : 205-8. 1943.

Progress in the use of molecular distillation is cited as the reason for compilation of this Supplement. Abstracts of references listed in previous bibliographies were published by Detwiler (Detwiler, S. B., Abstracts of articles and patents on molecular or short-path distillation. U. S. Dept. Agr. Bur. Agr. Chemistry Eng. ACE-115, 98 pp, 1941, (mimeographed.) U. S. Reg. Soybean Ind. Prod. Lab., Urbana, Ill.) The present supplement contains approximately 190 citations, including earlier articles and patents not listed in first two bibliographies. Where available, the Chemical Abstract reference has been included with each citation.

40. COLORED CHROMATOGRAMS WITH HIGHER FATTY ACIDS. Graff, M. M.; and Skau, E. L. Ind. Eng. Chem. 15 : 340-41. 1943.

A method has been described for separating mixtures of higher fatty acids by Tswett adsorption analysis, in which separation into zones was observed on a column of heavy magnesium oxide impregnated with a suitable indicator. The fatty acids were recovered by dissolving the magnesium oxide in particular sections of the column in acid and extracting with ether. By this procedure it was possible to demonstrate a separation of an unsaturated fatty acid from a saturated fatty acid of the same number of carbon atoms and of two saturated fatty acids differing in chain length by four carbon atoms.

37. LABORATORY DEODORIZER FOR FATS AND OILS. Bailey, A. E.; and Feuge, R. O. Ind. Eng. Chem. 15: 280-81. 1943.

A simplified apparatus to deodorize fats and oils in the laboratory is described, with illustrative diagram.

32. SMOKE, FIRE, AND FLASH POINTS OF COTTONSEED, PEANUT, AND OTHER VEGETABLE OILS. Morgan, D. A. Oil & Soap 19: 193-98. 1942.

Smoke, fire, and flash points of a variety of cottonseed and peanut oils have been determined, and effects of free fatty acid content, processing treatments, and prolonged heating at 350° F. on these properties investigated. The smoke points of good quality cottonseed and peanut oils

processed under similar conditions differ little, but this difference is believed to be significant. Presence of free fatty acids in either cottonseed or peanut oils influences the smoking temperatures and appears to be the principal, but not the only factor that tends to lower the smoke point. Results indicate that the other substance (or substances) other than free fatty acids, partly responsible for lowering the smoke point, is more or less unsaturated. The type of unsaturated bodies in cottonseed and peanut oils that may account for this phenomenon is exemplified by the carotenoid pigments.

MEAL - COMPOSITION, PROPERTIES, SPECIAL PRODUCTS AND PROCESSES

3062. RESEARCH ACCOMPLISHMENTS WITH SEED PROTEINS FOR HUMAN FOOD. Altschul, A. M. Natl. Res. Council. Agr. Res. Inst. Proc., 14th Ann. Meeting: 41-50. 1965. (No reprints available.)

In the face of a growing deficiency of protein concentrates for human food, there is increasing awareness of the possibility of seed proteins filling part of this gap. Many seeds, particularly the oilseeds, have sufficient protein content so that by relatively simple processing operations, products containing 50 percent or more protein may be obtained. By further manipulations, protein contents up to 100 percent may be achieved. These protein concentrates can be so produced that they are nutritionally adequate, safe, reproducible, and satisfying. This paper outlines the properties that these products should have, information already available for immediate application, and the types of information needed to broaden the use of seed protein concentrates for human food. Their nutritional value is discussed and the limiting amino acids are identified. Methods for measuring adequacy of the protein product and the extent of heat damage during processing are discussed, as well as methods for measuring the removal of interfering materials. Progress has been made in basic research on seed proteins: two areas discussed are the subcellular location of the major proteins of seeds and improvement in methodology towards obtaining pure seed proteins. In view of the information already obtained and the research capability achieved, there is reason to believe that seed protein concentrates can now be utilized in places where there is a protein shortage; further research and development to extend their utilization can be accelerated because new basic tools and approaches are now available.

3056. A LOOK AT THE WORLD PROTEIN SITUATION. Altschul, A. M. Oil Mill Gaz. 70(6): 14-20. 1965.

Protein deficiency in underdeveloped countries is a major problem facing the world today. Increased production of seed proteins (cottonseed, peanuts, and soybeans, for example) could close the protein gap in countries where animal protein is scarce. Problems relating to utilization of the nutritive values of seed proteins and to acceptance remain to be solved. Seed proteins as new sources of protein concentrates could fill the need in protein-hungry countries if the problems are attacked with new ideas and new approaches.

3050. COTTONSEED PROTEINS: THEIR STATUS IN NONRUMINANT FEEDING. Frampton, V. L. Cereal Sci. Today 10: 577-82, 589. 1965.

There is wide variability in the nutritive quality of cottonseed meals for nonruminants. This wide variability is largely accounted for by the variation in amount of available lysine in the meals. Lysine of cottonseed proteins, with the epsilon amino groups bound chemically, is not used by broilers even though they are under severe protein stress. The variance of total gossypol in cottonseed meals does not make a measurable contribution to the total variance in

growth of chicks receiving cottonseed meals in practical rations, and the correlation of growth of chicks with free gossypol is virtually zero, although the level of free gossypol may range up to 0.3 percent. High levels of available lysine in cottonseed meals are obtained when cooking is avoided in the processing of cottonseed and when the oil and most of the gossypol are removed from raw moist flakes with a solvent mixture composed of hexane, acetone, and water. This solvent mixture forms an azeotrope (49° C.) that is an excellent solvent for oil and gossypol. Quantitative extraction of oil from raw moist cottonseed flakes can be obtained through the use of this solvent in a matter of minutes. The resultant meals are on par with toasted soybean meals in cereal-based rations for nonruminants.

2983. EDIBLE SEED PROTEIN CONCENTRATES: THEIR ROLE IN CONTROL OF MALNUTRITION. Altschul, A. M. Israel J. Med. Sci. 1(3): 471-79. 1965.

The problem of protein malnutrition is discussed. The paper points out the partnership between the medical and food sciences in evaluation and solution of this problem, and is illustrated by consideration of the possibility of increasing the world edible protein supply by exploiting present technology for potential development of protein concentrates from seeds.

2950. STUDIES ON THE EXTRACTION AND PRELIMINARY CHARACTERIZATION OF COTTONSEED PROTEINS (PL-480 GRANT). Mondovi, B.; Fasella, P.; Turano, C.; Wolf, A. M.; Scioscia Santoro, A.; Turini, P.; Bossa, F.; and Cavallini, D. (Instituto di Chimica Biologica dell'Universita di Roma). J. Biochem. (Italian) 13: 413-19. 1964.

The proteins of cottonseed have been extracted by various procedures to obtain preparations in which the protein fractions underwent the smallest possible structural and functional changes. By means of thin-layer chromatography, the presence of three protein fractions was demonstrated, two having a greater and one a smaller mobility than ox serum albumin.

2625. DETERMINATION OF AVAILABLE LYSINE IN OILSEED MEAL PROTEINS. Rao, S. R.; Carter, F. L.; and Frampton, V. L. Anal. Chem. 35: 1927-30. 1963.

A simple and rapid method for estimating available lysine in plant proteins is described. The protein is dinitrophenylated and hydrolyzed in acidic media. The epsilon-DNP-lysine produced is eluted from an ion-exchange column with a solvent composed of three parts by volume of 3N aqueous HCl and one part methyl ethyl ketone, and is determined spectrophotometrically at 435 mμ.

2408. ISOLATION AND CHROMATOGRAPHIC CHARACTERIZATION OF LOW MOLECULAR WEIGHT COTTONSEED PROTEINS. Martinez, W. H.; and Frampton, V. L. J. Agr. Food Chem. 10: 412-15. 1962.

Glandless cottonseed meal fractionated by successive extraction with aqueous ethanol solutions of varying salt content yielded three fractions. Each is high in nitrogen and sulfur and low in ash and contains nonreducing carbohydrates. Each gives a single peak in the ultracentrifuge with an estimated S_{20} value of 2 but shows a multicomponent system when analyzed with cation-exchange cellulose chromatography. Analysis showed the fractions to differ in content from whole meal and each other, particularly in such nutritionally important amino acids as lysine, leucine, cystine, and glutamic acid.

2286. EVALUATION OF COTTONSEED MEALS PREPARED BY EXTRACTION WITH ACETONE-HEXANE-WATER MIXTURES. Mann, G. E.; Carter. F. L.; Frampton, V. L.; Watts, A. B.; and Johnson, Charles. J. Am. Oil Chemists' Soc. 39: 86-90. 1962.

Eleven cottonseed meals have been prepared by batch extractions of a given lot of cottonseed with various acetone-hexane-water mixtures using several different extraction schedules.

These meals, together with eight meals of commercial origin and a commercial soybean meal, have been subjected to chemical evaluation and assayed for protein quality using the growing chick as a test animal. In general the acetone-hexane-water meals were superior to the commercial cottonseed meals for promoting the growth of the chicks and, considering all the meals, a linear correlation was obtained between the logarithm of the weight gains and the available lysine contents of the meals. The free and total gossypol contents of the cottonseed meals appeared to have little or no influence on the growth rates.

2279. COTTONSEED CONSTITUENTS AND DISCOLORATIONS IN STORED SHELL EGGS. Frampton, V. L.; Carter, F. L., Piccolo, Biagio; and Heywang, B. W. J. Agr. Food Chem. 10: 46-48. 1962.

Frequently whites of eggs from hens fed cottonseed meal develop a pink color during storage. This color change is directly correlated with the Halphen positive constituent of the meal. This constituent induces an increase in the pH of the yolk during storage of shell eggs. The development of brown color in the yolk of stored shell eggs produced by hens fed cottonseed meal is enhanced by the presence of a Halphen-positive constituent since the chromogen responsible for the brown color is pH-sensitive.

2274. LYSINE, GOSSYPOL, AND NITROGEN SOLUBILITY IN CHEMICALLY TREATED COTTONSEED MEALS. King, W. H.; Kuck, J. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 39: 58-60. 1962.

The effects of treatment of commercial prepress-solvent extracted and direct solvent-extracted cottonseed marcs with several chemical agents and solvents were studied. The analytical results for "free" and "total" gossypol of the finished meals show that treatment with aliphatic amines, followed by extraction with a suitable solvent, removed large proportions of the "free" as well as "bound" gossypol. This reduction of "free" and "bound" gossypol was accompanied in some experiments by an increase in the nitrogen solubility and available lysine, as compared with the results obtained with the untreated air-dried marcs. The available lysine contents of the treated marcs was significantly correlated with the nitrogen solubility in 0.02 N aqueous NaOH.

2227. MICROBIOLOGICAL ASSAY OF INTACT PROTEIN USING TETRAHYMENA PYRIFORMIS W. I. SURVEY OF PROTEIN CONCENTRATES. Teunisson, D. J. Anal. Biochem. 2: 405-20. 1961.

A survey was made of the relative nutritional quality of 10 protein concentrates and of a mixture of amino acids determined by assay with Tetrahymena pyriformis W. I. The method is essentially that of Rosen and Fernell, with the simplifying modification proposed by Rosen in 1958 except that utilization of the nitrogen source was evaluated by growth response curves and lyophilized mixtures of some of the basal medium components were employed. The soundness of the counting procedure used in estimating this growth response was investigated. The reproducibility of the result at several nitrogen levels of the reference standard, "vitamin-free" purified casein, was established by statistical studies. Nutritional quality is estimated as an index: the ratio of the regression coefficient of growth on the nitrogen content of the test material to the regression coefficient of growth on the nitrogen content of casein. These were estimated for the ranges 0 to 1.0 and 0 to 1.7 mg. nitrogen/ml. These ranges were adopted because there was better growth at these concentrations for most of the nitrogen sources studied. Assays at higher nitrogen concentrations yield further information of the nutritional quality, particularly for diets containing mixed proteins. The same general response occurred with casein and most of the materials tested; growth increased with increasing nitrogen concentration up to a maximum and then decreased with higher nitrogen levels. Growth response curves are characteristic of each nitrogen source.

2192. PRESENT STATUS OF PROTEINS FROM OILSEEDS. Altschul, A. M. "Progress in Meeting Protein Needs of Infants and Preschool Children, Proc. Internat. Conf., Washington, D. C. Aug. 21-24, 1960" Natl. Acad. Sci.--Natl. Research Council Publ. 843: 517-30. 1961.

Principles and practices involved in obtaining plant protein mixtures of high nutritive value are discussed. Subjects are the quantity of oilseeds potentially available for incorporation into mixtures suitable for human foods; problems to be solved in order that these materials can be used effectively in human diets; opportunities already existing for incorporating these materials into high-protein diets; and what can be expected as knowledge and technology advances.

2175. AZEOTROPE EXTRACTION OF COTTONSEED. Frampton, V. L. Oil Mill Gaz. 66(2): 33-39. 1961.

Factors that impair the nutritive quality of commercially produced cottonseed meals are listed and identified. These include the destruction of lysine during processing, and binding of lysine by gossypol to yield meals that are deficient in lysine, the physiologically active bound gossypol that is responsible for egg yolk colors, the crude oil constituent that is responsible for pink egg whites, and the heat-labile antinutritional factor that is present in cottonseed. Many of the adverse factors may be reduced in intensity through the use of a solvent system composed of acetone, hexane, and water in the processing of cottonseed. Data given show that meals produced through the use of this solvent mixture are superior to presently produced commercial meals as a protein supplement for broilers, swine, and laying hens.

2095. COTTONSEED MEAL IN POULTRY FEED. COLLABORATIVE STUDY OF THE AGU METHOD OF GRADING COTTONSEED MEALS FOR LAYING RATIONS. Frampton, V. L.; and Piccolo, Biagio. J. Agr. Food Chem. 9: 129-31. 1961.

The results of a collaborative study of the available gossypol unit (AGU) method of grading cottonseed meals for laying rations indicate that AGU values differ significantly between various meals, and eggs and birds differ in their responses. Correlation between AGU values of cottonseed meals and coloration in yolks of stored shell eggs produced by the meals is virtually zero. The AGU method may not be relied upon for grading cottonseed meals for laying rations.

2066. NUTRITIVE QUALITY OF COTTONSEED MEALS. EFFECTS OF GOSSYPOL AND RAFFINOSE ON LYSINE CONTENT AND NUTRITIVE QUALITY OF PROTEINS IN MEALS FROM GLANDLESS COTTONSEED. Martinez, W. H.; Frampton, V. L.; and Cabell, C. A. J. Agr. Food Chem. 9: 64-66. 1961.

The relative importance of gossypol and raffinose in binding and destroying lysine and in impairing the nutritive value of cottonseed meals was studied with meals from glandless cottonseed. Nutritional evaluations with protein-depleted rats showed that: raffinose in cottonseed reduces lysine content and nutritive quality of the proteins when heat is applied; 1 percent concentration of gossypol is not as effective as 10 percent of raffinose in destroying lysine in cottonseed meal; gossypol and raffinose at these same concentrations are comparable in reducing the level of free epsilon-amino lysine in cottonseed proteins; and the nutritive index of cottonseed meals as determined by the rat repletion method is highly correlated with the free epsilon-amino groups of lysine of the protein and poorly correlated with total lysine.

2065. COTTONSEED MEAL IN POULTRY FEED. DISCOLORATIONS IN STORED SHELL EGGS PRODUCED BY HENS FED COTTONSEED MEAL. Frampton, V. L.; Piccolo, Biagio; and Heywang, B. W. J. Agr. Food Chem. 9: 59-63. 1961.

The chromogen that appears in yolks of eggs produced by hens fed rations containing cottonseed meal and causes the brown color to develop in such eggs (stored under refrigeration in the shell)

is a pH indicator. A brown color develops in an alkaline medium, and reverts to yellow when the medium is acidified. The relative chromogen concentration may be estimated photographically, with color film, where portions of the yolk are adjusted to pH 4. 6 and 10. 4. Difference in the transmittance spectra of the positive transparencies were used as a measure of relative chromogen concentration in the yolk. The color data correlate well with the intensity of coloration estimated visually. Correlations between intensity of coloration in the yolks and total gossypol, "free" gossypol, chemically uncombined gossypol, and gossypol-like pigments were all poor. Evidence indicates that the chromogen in cottonseed meals responsible for the brown coloration in stored shell eggs is heat stable.

2062. PROPERTIES OF COTTONSEED MEALS PREPARED WITH ACETONE-PETROLEUM ETHER-WATER AZEOTROPE. King, W. H.; Kuck, J. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 38 : 19-21. 1961.

Results are presented of a study of extraction of raw cottonseed meats with mixed slovents. A solvent mixture consisting of 53 percent acetone, 44 percent petroleum ether (boiling range 68° to 71° C., known in the vegetable oil industry as commercial hexane) and 3 percent of water, by volume, proved effective in extraction of oil, as well as gossypol, from the meats. The extracted meats had low gossypol content and retained the high epsilon-amino lysine content of the raw unextracted meats. The particular solvent mixture used was more effective in extraction of the gossypol and oil than were any of its components used alone. The mixture is easily recovered by continuous distillation from the oil and meal at a temperature lower than the boiling point of any of its components.

1940. A REPORT ON THE PROBLEM OF RESIDUAL SOLVENT IN SOLVENT-EXTRACTED MEALS. Gastrock, E. A.; Spadaro, J. J.; Crovetto, A. J.; and Brian, Ross. J. Am. Oil Chemists' Soc. 37 : 192-95. 1960.

The hazards of excess residual solvent in solvent-extracted meals and the methods currently used for its measurement are discussed. Preliminary results are reported with a simple copper-cup flash tester with concentric rings as heating surfaces. Solvent contents as low as 0. 03 percent can be detected. The method is semiquantitative. The method may be useful for plant-control purposes.

1764. ADVANCES IN THE DETERMINATION OF AMINO ACIDS IN FEEDSTUFFS. Frampton, V. L. Feedstuffs 31(25) : 18-20. 1959.

It may be said that the chromatographic method for the determination of amino acids present in protein hydrolyzates is very good. The uncertainty in protein analyses, especially with feedstuffs where extraneous materials are present, develops because of destruction of amino acids during hydrolysis and because of the unequal rates of hydrolysis of the peptide bonds. The preparation of the amino acid solution by the hydrolysis on the protein should by given concentrated research attention in the immediate future.

1627. SOLUBILITY OF COTTONSEED PROTEINS IN HYDROCHLORIC ACID. Mann, G. E.; Rubins, R. K.; Carney, W. B.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 35: 244-46. 1958.

The correlations between the growth response of chicks to the nine cottonseed meals fed as a protein supplement and the solvent powers of 0. 02N NaOH, 6NHC1, and 0. 5N NaC1 for cottonseed meal proteins are almost identical. The correlations between the solvent power of 0. 02N NaOH, 6N HC1, and 0. 5N NaC1 and the gossypol contents of the meals are as good as the correlations between the solvent powers of these solutions and the growth response of chicks.

1621. PILOT-PLANT DEVELOPMENT OF THE ALKALI-COOKING PROCESS FOR COTTONSEED MEATS. III. QUANTITATIVE EFFECT OF COOKING VARIABLES ON SOLUBILITY OF MEAL NITROGEN. King, W. H.; Knoepfler, N. B.; and Hoffpauir, C. L. J. Am. Oil Chemista' Soc. 35: 46-49. 1953.

Comparison of data from experimental cooking of cottonseed under closely controlled conditions of temperature, moisture, and time showed that protein denaturation, as measured by the solubility of the meal nitrogen in 0.02N NaOH, closely followed the equation for a first-order reaction with a velocity constant of 0.012. Laboratory data obtained under conditions simulating commercial practice permitted the development of an empirical relationship between the reduction of nitrogen solubility and moisture, temperature, and time. Mathematical treatment of these data led to an empirical formula relating cooking conditions to protein denaturation, which correlated well with analytical data. The formula indicates that the temperature increment above 135° F. and the cooking time in minutes are equally effective. When these variables are added and the sum is multiplied by a moisture factor, consistent values commensurate with the reduction in nitrogen solubility of the meal are obtained.

1612. LYSINE CONTENT OF COTTONSEED MEALS. Martinez, W. H.; and Frampton, V. L. J. Agr. Food Chem. 6: 312. 1953.

To study the sensitivity of lysine in cottonseed to destruction during processing, a series of seeds and commercially produced meals were analyzed for lysine content by means of a modified Moore and Stein ion-exchange procedure. A decrease in the lysine content is noted when cottonseed is processed for oil and meal. The extent of this decrease depends upon the severity of the process and not upon the type of process used. It is possible that part of the variation in the growth response of animals fed cottonseed meal as a protein supplement may be accounted for on the basis of the variation in the lysine supplied by the meal.

1455. NOTE ON ACETONE-SOLUBLE MATERIAL IN COTTONSEED MEALS. King, W. H.; Frampton, V. L.; and Altschul, A. M. J. Am. Oil Chemists' Soc. 34: 247-49. 1957.

In connection with a study of methods of eliminating traces of free gossypol from cottonseed meals, a procedure has been developed for isolation of gossypol from the 70 percent acetone extracts of the meals by transfer to benzene solution. A procedure is also given for quantitative determination of the isolated gossypol. Analysis of four samples of cooked cottonseed meats and meal by the method showed that in each instance substances other than gossypol are measured by the A. O. C. S. method for free gossypol in meals containing low concentrations of residual free gossypol.

1453. CHANGES INDUCED BY AUTOCLAVING A SOLVENT-EXTRACTED COTTONSEED MEAL. Conkerton, E. J.; Martinez, W. H.; Mann, G. E.; and Frampton, V. L. J. Agr. Food Chem. 5: 460-63. 1957.

A study was made of the effect of autoclaving a prime quality solvent-extracted meal upon its soluble protein fractions. Portions of both the original and the autoclaved meal were fractionated successively with water, saline solution, and alkali. In addition to modifying the solubility behavior of the cottonseed protein, autoclaving reduced the sugar, lysine, and arginine contents of the meal. From these results it is apparent that the simple measurement of the solubility of nitrogen in the meal, as commonly used to indicate the degree of heat damage to a meal, would not reflect the complete change induced in cottonseed protein by heat. An extension of this type of study to commercially produced meals may provide a more thorough understanding of the effects of different types of processing on the chemical constituents of the meals and their relationship to nutritive quality.

1231. STATUS OF RESEARCH ON CHEMICAL METHODS OF MEASURING NUTRITIVE VALUE OF COTTONSEED MEAL. Altschul, A. M. Oil Mill Gaz. 60(5): 10-12. 1955.

The place of cottonseed meals as a protein supplement for ruminants and for nonruminants such as poultry and swine is discussed briefly. Free gossypol is generally accepted as a reliable index to toxicity of cottonseed meal in the feeding of poultry and swine, and effects of various methods of processing on the free gossypol content are outlined. Nitrogen solubility tests are widely used to measure nutritive value. High temperatures during processing tend to reduce nitrogen solubility. The paper is a review of the status and prospects of research on cottonseed as a protein supplement for feeding of nonruminants.

1108. EFFECT OF AUTOCLAVING IN PRESENCE AND ABSENCE OF GOSSYPOL ON SOLVENT-EXTRACTED COTTONSEED MEAL. Condon, M. Z.; Jensen, E. A.; Watts, A. B.; and Pope, C. W. J. Agr. Food Chem. 2: 822-26. 1954.

Before the processing conditions in cottonseed oil mills can be modified to produce meals of consistently high nutritional quality the individual effects of heat and of bound gossypol must be understood. This investigation was undertaken to determine the effect of autoclaving in the presence and absence of gossypol on certain chemical and nutritive properties of a cottonseed meal of initially high quality. Chick feeding experiments indicated a progressive decrease in the protein quality index of the meals as time of autoclaving increased. This reduction was paralleled by similar decreases in solubility of nitrogen of the meals in 0. 02N sodium hydroxide. There appeared to be no direct relation between any other property of the meals and their nutritive value to chicks. Samples to which 1 percent gossypol was bound during autoclaving appeared to be equivalent, chemically and nutritionally, to control samples autoclaved for the same time.

1030. RESEARCH ON COTTONSEED MEALS. Jensen, E. A.; and Altschul, A. M. Cotton Gin & Oil Mill Press 55 (11): 16, 18, 50-51. 1954.

Most important result of an investigation of the effects of autoclaving on the nutritive and the chemical and physical properties of cottonseed meals was the indication of a strict parallelism between reductions in protein quality index and decreases in nitrogen solubility, suggesting that individual components of the protein (amino acids) are altered in some way during processing, either by heat alone or by reaction with other meal constituents under the influence of heat. Time of autoclaving was the main cause of reductions of nutritive value of a standard meal (one of low gossypol content and high nutritive value, prepared by successive extractions with hexane and butanone). The next objective will be to establish methods for determining the amino acids content of cottonseed meals and meals fractions, as a basis for relating changes in content to changes in nutritive value.

1028. THE EFFECT OF PROCESSING CONDITIONS ON THE NUTRITIVE VALUE OF COTTONSEED MEAL FOR POULTRY--A REVIEW. Altschul, A. M. Poultry Sci. 33: 180-185. 1954.

This article summarizes cooperative research underway to develop means of producing, economically and regularly, cottonseed meals that will be suitable for unrestricted use in poultry feeds. The program comprises laboratory research on the chemistry of cottonseed processing; development of a chemical measure of nutritive value; practical experiments in industrial mills; and cooperative experiments with nutrition investigators. Feeding tests show that screw pressing can produce meals that are safe for unrestricted use in poultry feeds under such conditions that the temperature of the meats entering the press is 200° F. or less. Also, some prepress solvent-extracted meals produced in commercial mills and not heated above 200° F. before pressing have free-gossypol contents of 0. 03 percent.

1015.1 NUTRITIVE VALUE OF BREAD AND COOKIES CONTAINING COTTONSEED FLOUR. Womack, Madelyn; Marshall, M. W.; and Summers, J. D. J. Agr. Food Chem. 2 : 138-40. 1954.

Water bread containing 10 parts of cottonseed flour per 100 parts of wheat flour (white) gave significantly higher rates of gain per gram of nitrogen consumed than water bread without the cottonseed flour when fed to young rats at levels furnishing 10 percent protein. When the effect of the addition of cottonseed flour to bread containing nonfat milk solids was tested, it was found that no significant increase in the nitrogen efficiencies was brought about by the cottonseed flour when the breads were both fed at the same protein level. However, when the breads were fed at the same percentage by weight, the higher protein content of bread containing cottonseed flour brought about significantly higher rates of gain than did the same amount of bread without cottonseed flour.

919.1 EXPERIMENTS CONDUCTED IN BAKING DEPARTMENT OF OKLAHOMA AGRICULTURAL AND MECHANICAL COLLEGE, SCHOOL OF TECHNICAL TRAINING. Summers, J. C.; Mead, Byron; and Thurber, F. H. Oklahoma Agr. Mech. Coll. Pamphlet, June 13, 1951, to June 13, 1953 Inc. 32 pp. Undated.

This pamphlet contains a collection of recipes developed for the use of cottonseed flour in the preparation of baked goods, including several types of bread and cake. The recipes are on a bulk scale for commercial or institutional use. The work was carried out under contract with the Southern Regional Research Laboratory to investigate the use of specially milled, 57 percent protein cottonseed flour in baked foods for human consumption, primarily for the purpose of increasing nutritive value through the increased protein. In the foreword the authors report that these foods were offered for sale in the college food store and cafeteria over an 18-month period. No evidence of allergies or other disturbances was observed, and baked foods with and without cottonseed flour were equally acceptable.

888. COTTONSEED MEAL EXTRACTS. ELECTROPHORETIC PATTERNS OF BUFFER EXTRACTS OF DIFFERENT NUTRITIVE VALUE. Karon, M. L.; Adams, M. E.; and Altschul, A. M. J. Agr. Food Chem. 1 : 314-18. 1953.

To determine the nature of changes taking place in cottonseed meal proteins as a result of differences in conditions of preparation, the electrophoretic patterns of cottonseed meal extracts were studied. As heat stress increased during processing, electrophoresis patterns of the soluble protein fraction of the meal changed. A new fast moving component developed, and the original two major components became more difficult to resolve. These changes seem to correlate well with changes in nutritive value within a single series of related meals.

887. RESEARCH ON COTTONSEED MEALS. Jensen, E. A.; Condon, M. Z.; Karon, M. L.; and Altschul, A. M. Cotton Gin & Oil Mill Press 54 (5) : 24-25, 28. 1953.

Data obtained by the National Cottonseed Products Association Fellow in research to develop a chemical measure of the nutritive value of cottonseed meal are reported. Changes in chemical and physical properties caused by autoclaving of a cottonseed meal of known high nutritive value are indicated. There is no change in the total or acid-soluble phosphorus or total nitrogen content, but free gossypol content and protein solubility in sodium chloride after autoclaving for 15 minutes decrease sharply. Inorganic phosphorus gradually increases as time of autoclaving increases. Protein solubility in sodium hydroxide, total gossypol content, and soluble carbohydrate content decrease as time of autoclaving increases. Electrophoretic patterns indicate progressive degradation of protein as time of autoclaving increases. Correlation of these data with the nutritive value of the meals must await the completion of chick feeding experiments being done by cooperating organizations.

880. A PRELIMINARY STUDY OF THE EFFECT OF MOISTURE CONTENT, ROLLING, AND COOKING OF COTTONSEED MEATS ON THE CHEMICAL PROPERTIES OF HYDRAULIC-PRESSED MEALS. Reuther, C. G., Jr.; LeBlanc, M. F. H., Jr.; Batson, D. M.; and Knoepfler, N. B. J. Am. Oil Chemists' Soc. 30 : 28-32. 1953.

Closely set five-high rolls, corrugated rolls, and smooth rolls were used to accomplish gland breakage in cottonseed meats, and their effectiveness was compared. The degree of gland breakage was determined by an empirical method using hexane extraction. Severe (five-high rolls) conditions resulted in large amount of gland breakage. A maximum gland breakage of 54 percent was generally obtained at moisture contents of 14 percent or above in the meats. Samples having a larger amount of gland breakage had a much lower content of free gossypol after cooking and pressing. Under the best conditions of rolling and cooking, in which the temperature of cooking did not exceed 225° F., a hydraulic-press meal was obtained with a free-gossypol content of 0. 03 percent and nitrogen solubility of 40 percent.

821. THE EFFECT OF TEMPERATURE ON THE ELECTROPHORETIC ANALYSIS OF COTTONSEED MEAL EXTRACTS. Karon, M. L.; Adams, M. E.; and Newman, S. J. Colloid Sci. 7 : 407-13. 1952.

The electrophoretic patterns of a cottonseed meal extract in the ethylamine barbital buffer (pH 10. 4 at 25° C.) have been investigated over the range of 0° to 20°. The results indicate that there is no change in the relative concentrations of the components as a function of temperature. The application of a single viscosity correction to the solvent could not entirely compensate for the change in mobility of each component with temperature. The viscosity-mobility product decreased with increasing temperature. This trend varied irregularly for the several components present.

753. COTTONSEED AND PEANUT MEAL GLUES. RESISTANCE OF PLYWOOD BONDS TO CHEMICAL REAGENTS. Hogan, J. T.; and Arthur, J. C., Jr. J. Am. Oil Chemists' Soc. 29 (1) : 16-18. 1952.

The resistance to both organic and inorganic reagents of birch plywood glue bonds using cottonseed or peanut meal or casein was determined for periods ranging from 1 to 14 days. It was suggested that the principal attractive forces involved in the protein bonds were ionic or valence forces and that differences observed in the resistance of the glues to the chemical reagents were probably due to variations in the amino acid constitution of the protein.

752. COTTONSEED MEAL AS A TIRE CORD-TO-RUBBER ADHESIVE. Hoffman, W. H., Jr. and Arthur, J. C., Jr.; Rubber Age 71 : 354-56. 1952.

Methods of dispersing cottonseed meal to yield a good adhesive of the cord-to-rubber type have been investigated and the adhesive properties of cottonseed meal have been compared with those of casein. On a laboratory scale, dips containing solvent-extracted cottonseed meal compared favorably with those containing casein. The viscosities of the former were higher than the latter, but were within a usable range for application to the cord.

654. VISCOSITY OF COTTONSEED MEAL DISPERSIONS. Hogan, J. T.; and Arthur, J. C., Jr. J. Am. Oil Chemists' Soc. 28 : 436-38. 1951.

It has been shown that the apparent viscosities of cottonseed meal dispersions depend on the concentration of meal and of sodium hydroxide, and on the addition of trichloroacetate ion. Also, it has been shown that the viscosities of the glue dispersions are independent of the nitrogen solubilities of the protein contained in the meal and, for the processes evaluated, independent of the method of removal of the oil to produce the meal.

643. LIST OF PUBLICATIONS AND PATENTS 1944-1950 ON COTTONSEED AND PEANUT PROTEINS AND RELATED SUBJECTS. Anonymous. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-314. 1951. (Processed)

Greater knowledge of the chemistry of cottonseed and peanut proteins would provide a reliable basis for developing new uses or expanding their present uses. This list of publications and patents for 1944-50 represents the initial accomplishment of the Southern Regional Research Laboratory in a program of research on the chemistry of these products. The references, 71 in all, are listed alphabetically by authors, within year divisions. An index is appended.

581. GETTING PROTEIN FROM COTTONSEEDS. Arthur, J. C., Jr.; and Karon, M. L. Yearbook Agr., U. S. Dept. Agr. 1950-51 : 619-20.

Cottonseed meal and cake, of which the United States produces about two million tons annually, are a source of raw materials for making adhesives and protein fibers. The production of plywood glue, plastic compounds, and fire-extinguishing liquids from cottonseed meal mixed with other ingredients is described. The method of producing cottonseed protein is given. The first step is to produce an oil-free meal, and the two general methods of accomplishing this, mechanical pressing and solvent extraction, are described. The six steps necessary to extract the protein from the oil-free meal are outlined. The general process for producing a fiber from cottonseed protein is given, and the potential value of the resulting fiber as a supplement to domestic wool supplies is mentioned.

553. COTTONSEED AND PEANUT MEAL GLUES: PERMANENCE OF PLYWOOD GLUE JOINTS AS DETERMINED BY INTERIOR AND EXTERIOR ACCELERATED CYCLIC SERVICE TESTS. Hogan, J. T.; and Arthur, J. C., Jr. J. Am. Oil Chemists' Soc. 28 : 272-74. 1951.

Data on the strength properties of cottonseed and peanut meal glues in plywood bonds as they are affected by accelerated interior and exterior cyclic service tests are reported. Cottonseed meal glue was superior to peanut meal glue and compared favorably with commercial casein glue on an interior test basis for five cycles. Cottonseed meal glue can probably be used for bonding plywood designed primarily for interior uses and for use in regions of comparatively low relative humidity. Cottonseed meal glue should be cheaper than casein glue on both a pound and glue line basis.

552. PREPARATION AND UTILIZATION OF COTTONSEED MEAL GLUE FOR PLYWOOD. Hogan, J. T.; and Arthur, J. C., Jr. J. Am. Oil Chemists' Soc. 28 : 20-23. 1951.

The preparation of plywood glues using hydraulic, pressed, screw-pressed, and hexane-extracted cottonseed meals is described; processing information is given for the preparation of plywood using the glue; and shear strength of the glue joints is reported. Hexane-extracted cottonseed meal glue compared favorably with commercial casein glue and peanut meal glue, each glue mix and glue line being prepared as recommended to give maximum shear strengths. Values of shear strength and wood failure of glue joints containing hydraulic- and screw-pressed meals, as compared to hexane-extracted meal, were lower.

545. CHEMICAL PROPERTIES AND NUTRITIVE VALUE OF COTTONSEED MEAL AS RELATED TO CONDITIONS OF PROCESSING. Altschul, A. M. Proc. 55th Ann. Conv. Natl. Cottonseed Prods. Assoc. 1951: 32-34, 36 (Publ. Cotton Gin & Oil Mill Press. Unnumbered.)

Research in cooperation with industry and with nutrition investigators shows that cottonseed meal is a variable nutritional material. The value of cottonseed meal as a feedstuff depends upon the type of animal fed, the amino acid content of the component protein, the damage to the protein that has taken place during processing, and the presence of any associated materials that would interfere with growth and food utilization. Experimental meals prepared

by the screw-press method have been fed to hogs and chicks in varying quantities and have supported good growth. Conditions of cooking before removal of oil in the screw press greatly affect the protein value of the meal. Experimental screw-press meals did not cause egg yolk discoloration when fed to laying hens.

438. INTRINSIC VISCOSITY OF COTTONSEED GLOBULIN SOLUTIONS. Arthur, J. C., Jr.; and Saik, B. H. J. Colloid Sci. 5 : 326-30. 1950.

The shape characteristics of cottonseed globulin molecules in solution as determined by viscosity data are important in adapting the globulin for use in making synthetic fibers or adhesives and in evaluating chemical and physical modification of the globulin. Globulin (18.06 percent nitrogen) was extracted from solvent-extracted cottonseed meal by means of 0.5 N sodium chloride solution, precipitated by dialysis, and lyophilized. The intrinsic viscosity of the globulin in urea solutions was calculated to be 0.093m equivalent to a viscosity increment of 12.3 and an axial ratio (Simha equation) of 9.4 assuming elongated macromolecules or of 16.3 assuming flattened-disc-type macromolecules.

452. ELECTROPHORETIC ANALYSIS OF PEANUT AND COTTONSEED MEALS AND PROTEINS. Karon, M. L.; Adams, M. E.; and Altschul, A. M. J. Phys. & Colloid Chem. 54 : 56-66. 1950.

Cottonseed and peanut meals and the derived proteins were analyzed by means of the Tiselius electrophoresis apparatus. The effect of the method of extraction of the protein and of the buffer and pH of the buffer solutions was investigated. By fractional precipitation involving a change of ionic strength the two major components of cottonseed protein can be concentrated to over 80 percent purity. Approximately 75 percent of peanut protein consists of two components. These migrate as a single entity unless the meal has been prewashed to remove soluble sugars and phytin, in which case the two major components separate into two almost equal fractions.

449. EFFECT OF PROCESSING CONDITIONS ON THE CHEMICAL PROPERTIES OF COTTONSEED MEALS. Haddon, R.; Schwartz, A. K.; Williams, P. A.; Thurber, F. H.; Karon, M. L.; Dechary, J. M.; Guice, W. A.; Kupperman, R. P.; O'Connor, R. T.; and Altschul, A. M. Cotton Gin & Oil Mill Press 52(9) : 18-20. 1950.

As part of research to improve the nutritional value of cottonseed meal by improving the methods of processing, the changes in chemical properties of cottonseed meals caused by variation in conditions of hydraulic- and screw-press operation were investigated. The processing experiments were conducted in a commercial mill on the effect of conditions of cooking the meats and of the degree of pressure exerted in the screw press. Changes taking place in the cottonseed protein were measured by determination of nitrogen solubility. The disposition of gossypol and gossypol-like compounds was determined by measurement of content of free-gossypol-like materials in the meal. In screw-press operations it was possible to obtain meals whose analysis showed low gossypol contents with 40 percent of their nitrogen soluble in half-molar salt solution. Collaborative feeding tests are underway for the purpose of correlating laboratory data on these properties with nutritive properties.

425. PILOT-PLANT FRACTIONATION OF COTTONSEED. II. DIFFERENTIAL SETTLING. Vix, H. L. E.; Spadaro, J. J.; Murphy, C. H., Jr.; Persell, R. M.; Pollard, E. F.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 26 : 526-30. 1949.

A new fractionation process termed "differential settling" has been developed to produce a cottonseed meal substantially free of oil, pigment glands, and hulls from either defatted or undefatted flakes. Work on this process was initiated during research on a mixed solvent-

flotation method of fractionation which showed several inherent disadvantages but which produced cottonseed meal essentially free of pigment glands (gossypol content as low as 0.006 percent). Such meal has high nutritional value and excellent potentialities for industrial use. Meal produced by the differential settling process has a gossypol content as low as the meal produced by the flotation principle and the process overcomes the disadvantages of the latter method. Two methods, centrifugal and tube settling, have been developed that show promise for commercial use in combination with present solvent-extraction processes.

415. RESEARCH ON EFFECT OF PROCESSING ON THE NUTRITIONAL VALUE OF COTTONSEED MEAL. Dechary, J. M.; and Altschul, A. M. Oil Mill Gaz. 54 (2): 13-15. 1949.

In cooperation with the cottonseed industry and nutrition experts, the Southern Regional Research Laboratory has recently inaugurated extensive research to improve the nutritional value of cottonseed meal by the development of new methods or improvements in present methods of processing. The value of cottonseed meal as a feed depends not only on the quality of its protein but also on the presence or absence of materials that interfere with the growth of animals. The interfering materials are concentrated in the pigment glands and probably consist of gossypol and gossypol-like compounds. These materials can be bound to the proteins of the meal during processing so that they no longer interfere with the food value of the meal. Once the proper conditions for processing are defined and methods for production control developed, it should be possible for the cottonseed industry to produce a more uniform and far superior cottonseed meal.

409. COTTONSEED PROTEIN FIBER. Arthur, J. C., Jr.; and Many, H. G. Textile Research J. 19: 605-8. 1949. (Republished: Oil Mill Gaz. 54 (8): 55-57. 1950.)

Difficulties encountered in isolating protein from cottonseed meal, in dispersing the isolated protein in an aqueous medium in high concentration, and in preparing viscous and tacky dispersions suitable for use in the preparation of an artificial fiber are described, and the Southern Regional Research Laboratory's progress in solving these problems is outlined. Detailed procedures are given for the laboratory production of cottonseed protein fiber having dry strength of 0.6 to 0.7 g. per denier and a wet strength of 0.2 to 0.3 g. This fiber is yellow to orange and has a soft hand. Test data compared with that of other man-made protein fibers are included.

391. VISCOSITY OF COTTONSEED PROTEIN DISPERSIONS. Cheng, F. W.; and Arthur, J. C. Jr. J. Am. Oil Chemists" Soc. 26: 147-50. 1949.

Effects of various methods of preparing meal and of preparing cottonseed protein dispersions upon the viscosity characteristics of the dispersion were investigated. Tacky and viscous cottonseed protein dispersions can be prepared from proteins isolated from hexane-extracted meals, deglanded, hexane-extracted meals, and isopropanol-extracted meals by means of sodium hydroxide and trichloracetate ion. The addition of sugars increases the viscosity of the dispersions, and dextrose significantly decreases the rate of change in the relative viscosity. Proteins isolated from hexane-extracted meals seem to be less modified than those isolated from deglanded, hexane-extracted meals, as indicated by the higher viscosity and stability of the dispersions.

368. THE USE OF AMINIZED AND PHOSPHORYLATED COTTON FABRICS AS ION-EXCHANGE MATERIALS IN THE PREPARATION OF OIL SEED PROTEINS. Hoffpauir, C. L.; and Guthrie, J. D. J. Biol. Chem. 178(1): 207-12. 1949.

Protein preparations low in ash and phosphorus content may be made by the use of anion-exchange materials to increase the pH of oilseed-meal suspensions and cation-exchange materials to reduce the pH of the protein dispersion to the isoelectric range. Preparations very low in ash and phosphorus may be obtained by the alternate use of aminized cotton fabric and

phosphorylated cotton fabric until deionization is almost complete before separation of the protein curd. The method is also useful for the purification of protein preparations made by the usual methods. Although commercial ion-exchange resins may also be used, the results with the fabrics have been better. The fabrics are also more convenient and are readily recovered and regenerated.

349. NOTE ON EFFECT ON CHICK GROWTH OF FEEDING MEALS PREPARED FROM FLAXSEED AND COTTONSEED TREATED WITH AMMONIA PRIOR TO STORAGE. Altschul, A. M.; Karon, M. L.; and Schaefer, H. C. Poultry Science 27 : 408-10. 1948.

Samples of meal produced from cottonseed and flaxseed which had been treated with ammonia and stored several months before processing were evaluated as protein supplements in chick diets. Meal from ammonia-treated flaxseed was equal in value to commercial soybean and cottonseed meals and superior to untreated flaxseed meal, previously found to be detrimental to growing chicks when fed in levels of 4. 5 percent or more of the ration. The value of cottonseed meal was not affected by ammonia treatment of the seed.

315. THE NUTRITIVE VALUE OF COTTONSEED FOR CHICKS AS AFFECTED BY METHODS OF PROCESSING AND CONTENT OF PIGMENT GLANDS. Boatner, C. H.; Altschul, A. M.; Irving, G. W., Jr.; Pollard, E. F.; and Schaefer, H. D. Poultry Sci. 27 : 315-28. 1948.

For 6 weeks, one lot of chicks received a basal diet containing 13 percent of screw-pressed soybean meal, while similar groups were fed experimental diets in which cottonseed meals were substituted for the soybean meal, and gossypol and pigment glands were added in measured amounts to the basal soybean diet. Gland-free cottonseed flour and two types of cottonseed meal (hydraulic-pressed and uncooked diethyl ether-extracted) supported excellent growth. Markedly inferior growth resulted when the experimental diets contained pigment glands or meal extracted with hexane from uncooked cottonseed. Thus, it appears that the physiologically deleterious components of unprocessed cottonseed are segregated in the pigment glands. Pure gossypol added to the diet produced relatively little retardation in growth, and a poor correlation was found between the nutritive value of the various cottonseed products and their content of gossypol and gossypurpurin.

314. PREPARATION AND PROPERTIES OF COTTONSEED PROTEIN DISPERSIONS. Arthur, J. C., Jr.; and Karon, M. L. J. Am. Oil Chemists' Soc. 25 : 99-102. 1948.

Industrial utilization of cottonseed meal and protein has been retarded because the extracted proteins were not appreciably dispersible in aqueous alkali solutions below pH 11. 7. Methods for preparing nongelling dispersions of cottonseed proteins which have good tackiness and desirable viscosities for industrial use have been investigated. Dispersions were prepared with the aid of anions such as trichloroacetate, borate, citrate, and nitrate. Properties of isolated cottonseed protein depend on the method used to isolate it, the concentration of protein in the dispersion, the concentration of sodium hydroxide used to disperse the protein, and the addition of trichloroacetate ion to prevent gelation. Effects of the trichloroacetate ion on aqueous cottonseed protein dispersions are summarized in two tables and two charts. Apparatus used in the experiment is described in detail.

219. PROTEIN-PHYTIC ACID RELATIONSHIP IN PEANUTS AND COTTONSEED. Fontaine, T. D.; Pons, W. A., Jr.; and Irving, G. W., Jr. J. Biol. Chem. 164: 487-507. 1946.

The protein-phytic acid solubility relationship over a wide pH range has been determined for peanut and cottonseed meals, as well as for the corresponding dialyzed meals and isolated proteins, and the theoretical and practical significance of the acid relationship is discussed.

Some comparative data on soybean meal are included. A few of the characteristics of the phytase present in the meals are described. The data, illustrated with numerous solubility curves, establish that the naturally occurring phytic acid in seed meals is responsible for the suppression of the solubility of the seed-meal proteins at pH values below their isoelectric points. Phytic acid is a major impurity in isolated seed-meal protein preparations, the amount depending upon the methods of extraction and precipitation and the degree of purification. The phytate ion may influence the degree of clarity of certain seed extracts.

197. PEPTIZATION OF PEANUT AND COTTONSEED PROTEINS. EFFECT OF DIALYSIS AND VARIOUS ACIDS. Fontaine, T. D.; Irving, G. W., Jr.; and Markley, K. S. Ind. Eng. Chem. 38: 658-62. 1946.

Data are presented to compare the peptization behavior of the proteins of peanut and cottonseed meals, proteins of the corresponding dialyzed meals, and isolated proteins. Although the shapes of the pH-peptization curves for cottonseed and peanut meals differ, the response of the proteins to the removal of dialyzable meal constituents is the same in both cases. This shows that the presence of naturally occurring substances in both meals decreases markedly the peptizability of the meal nitrogen at certain acid pH values by exerts no effect at alkaline pH values. Data are given on the peptization behavior of the proteins of the peanuts and cottonseed preparations in relatively concentrated solutions of various acids. Hydrochloric and sulfuric acids exhibit the broadest working range of concentration for protein precipitation, 1.0 to 5.0 N. Monochloroacetic acid compares favorably at 1.0 to 3.0 N concentration, whereas, in general, acetic and phosphoric acids are poor protein precipitating agents. Trichloroacetic acid, although a slightly better protein precipitant than hydrochloric or sulfuric acid, is effective over a narrower range (0.5 to 2.0 N) of concentration.

144. IMPROVEMENT IN THE COLOR OF PEANUT AND COTTONSEED PROTEINS. Fontaine, T. D.; Detwiler, S. B., Jr.; and Irving, G. W., Jr. Ind. Eng. Chem. 37: 1232-36. 1945.

Protein preparations as light or lighter in color than commercial samples of soybean protein can be obtained from the meal of white-skin and blanched red-skin peanuts without the use of bleaching agents. The color of proteins prepared from meals of unblanched red-skin peanuts and cottonseed is improved considerably through the use of controlled protein extraction and precipitation techniques and by washing the moist protein precipitates with organic solvents such as dioxane, acetone, and methyl ethyl ketone. The protein preparations approach in color very close to the range of practical usefulness for the production of adhesives and fibers. The use of heat in drying is shown to exert no appreciable influence upon the color of dried peanut protein.

142. VISCOSITY PATTERNS OF PEANUT PROTEIN SOLUTIONS--COMPARISON WITH OTHER VEGETABLE PROTEINS AND WITH CASEIN. Burnett, R. S.; Roberts, E. J.; and Parker, E. D. Ind. Eng. Chem. 37: 276-81. 1945.

Methods for preparing vegetable protein solutions, such as peanut, cottonseed, and soybean, at useful concentrations and of determining their viscosity are described. Data presented show the influence of concentration, heat, pH, time and other factors on the viscosity relations of peanut protein solutions made alkaline with sodium hydroxide. These factors are, in turn, influenced by the conditions under which the peanut meal is prepared, by the methods employed in separating the protein from the meal, and by the subsequent treatment received by the separated protein. The viscosity behavior of peanut protein solutions is compared with that of solutions of casein and of soybean and cottonseed proteins.

121. LYOPHILIZATION APPARATUS. Pomes, A. F.; and Irving, G. W., Jr. Science 101: 22. 1945.

A simple, inexpensive lyophilization apparatus which has proved successful for moderately large-scale laboratory work is described and the considerations involved in its operation are discussed.

113. CONTROLLING MOISTURE CONTENT IN WOOD AND GLUED TEST SAMPLES. Burnett, R. S.; and Merrifield, A. L. Mech Eng. 67: 475-76. 1945.

An inexpensive, easily constructed, constant-humidity room is described for testing of joints made with water-resistant glues under standardized and reproducible moisture conditions. The specific temperature and humidity conditions for evaluating glue by plywood and block shear tests are tabulated.

82. THE REACTION OF AMMONIA IN THE VAN SLYKE VOLUMETRIC AMINO NITROGEN METHOD. Irving, G. W., Jr.; Fontaine, T. D.; and Samuels, C. S. Arch. Biochem. 4: 347-59. 1944.

The extent of reaction of ammonia with nitrous acid in the Van Slyke volumetric amino nitrogen method, in cases where the ammonia concentration lies within the range normally found in protein hydrolyzates, is measurable and significant. The fraction that reacts is independent of the initial concentration of ammonia and of the presence or nature of amino compounds, but depends upon the length of the reaction period and the structural characteristics of the apparatus used. An ammonia correction factor can be determined for any given apparatus and experimental conditions, and this factor can be applied to Van Slyke values obtained on protein hydrolyzates for the purpose of calculating "true" amino nitrogen values.

73. A DEVICE FOR RENEWING THE FILTER-CAKE SURFACE IN SMALL-SCALE VACUUM FILTRATIONS. Burnett, R. S.; and Merrifield, A. L. Ind. Eng. Chem. Anal. Ed. 16: 365. 1944

This device has been used successfully on a laboratory and semipilot-plant scale to expedite the vacuum filtration of alkaline dispersions of vegetable protein extracted from peanut and cottonseed meals. The principle of this laboratory filter cake scraper is similar to that used on one type of rotary drum filter, in that the clogged filter-aid surface is removed by scraping.

MEAL-SPECIAL PRODUCTS AND PROCESSES--PATENTS

2052. PRODUCTION OF COTTONSEED MEAL OF IMPROVED QUALITY. King, W. H.; and Frampton, V. L. U. S. Pat. No. 2,950,198. August 23, 1960.

A process for extracting cottonseed with a mixed solvent. The quality of the protein in the extracted cottonseed meal remains unimpaired since the improved extraction method avoids the use of heat.

1971. TREATMENT OF COTTONSEED MEALS TO REMOVE RESIDUAL PIGMENTS. King, W. H.; and Frampton, V. L. U. S. Pat. No. 2,934,432. April 26, 1960.

A process for treating cottonseed meals (after commercial solvent extraction) to remove residual gossypol and other biologically active pigments without damaging the cottonseed protein. Meals treated according to the process can be fed to laying hens without causing the production of discolored eggs.

1793. PREPARATION OF COTTONSEED MEAL SUITABLE FOR UNRESTRICTED USE IN LAYING HEN DIETS. King, W. H.; Altschul, A. M.; Dechary, J. M.; and Frampton, V. L. U. S. Pat. No. 2,873,191. February 10, 1959.

A process for treating cottonseed meats to provide meals suitable for feeding in unrestricted amounts to laying hens. The eggs produced by hens consuming cottonseed meal treated according to this process do not become discolored upon storage.

1792. DETOXIFYING COTTONSEED MEAL. King, W. H. U. S. Pat. No. 2, 873, 190. February 10, 1959.

A process for detoxifying cottonseed meal. Free gossypol can be reduced to amounts not exceeding 0. 01 percent in meals treated by this process.

1321. ACYLATED PROTEINS. Mann, G. E. U. S. Pat. No. 2, 729, 638. January 3, 1956.

Polypeptides such as proteins are converted to proteinaceous products of high intrinsic viscosity when acylated by the Schotten-Baumann procedure with polyhalides capable of reacting with a liquid alcohol under the conditions of the Schotten-Baumann procedure at a temperature below 30° C. An alcoholic hydrogen atom each is split out of at least two polyhalide halogen atoms until the intrinsic viscosity of the polypeptide product is markedly greater than that of the polypeptide starting material.

965. COTTONSEED MEAL GLUES OF LOW ALKALINITY. Arthur, J. C., Jr.; and Hogan, J. T. U. S. Pat. No. 2, 646, 408. July 21, 1953.

The process of producing cottonseed meal glues so slightly alkaline that they do not discolor thin light-colored veneers or acid woods. The glues are produced by dispersing a relatively high protein cottonseed meal in an aqueous solution consisting essentially of a water solution of urea and a lesser amount of formaldehyde, and maintaining the dispersion at from about 20 to 40° C. for about an hour, until the dispersion has the desired viscosity.

964. IMPROVED COTTONSEED MEAL GLUE. Arthur, J. C., Jr.; and Hogan, J. T. U. S. Pat. No. 2, 662, 023. December 8, 1953.

The improved glues are characterized by a high tack, good spreadability, and retention of a viscosity of about 200 poises for a relatively long working life of about 3 hours. They are produced by incorporating from about 1 to 2 percent (based on the weight of meal) of trichloroacetate anions into a conventional oilseed meal glue formulation in which the meal is cottonseed meal.

687. PROCESSES FOR THE FRACTIONATION OF COTTONSEED. Vix, H. L. E.; Spadaro, J. J.; Pollard, E. F.; Gastrock, E. A.; Persell, R. M.; and Murphey, C. H., Jr. U. S. Pat. No. 2, 579, 526. December 25, 1951.

Cottonseed meal is disintegrated in hexane to reduce at least 70 percent of the meal tissue to a size of 2 to 40 microns, the disintegration releasing the intact pigment glands. The meal is then separated by settling in a body of hexane, the settling being continued until the hulls and whole glands settle below and the firm particles of meal tissue remain suspended in the hexane. The suspension is then removed.

634. PROCESS FOR MANUFACTURING ARTIFICIAL FIBER FROM PROTEINS CONTAINED IN COTTONSEED. Arthur, J. C., Jr.; Karon, M. L.; Pomes, A. F.; and Altschul, A. M. U. S. Pat. No. 2, 462, 933. March 1, 1949.

A process by which the cottonseed protein is first treated with acid in a manner that partly denatures it, breaks the crosslinkages that interfere with the dispersion, and transforms the protein into a form from which dispersions can be made. The protein is extracted from gland-free cottonseed meal with dilute alkali; precipitated by the addition of acid and further treated with acid (present in concentrations of 0. 3 to 1. 0 molar); and dispersed in sufficient alkali to form a dispersion of from 4 to 150 poises. Such dispersions have been successfully spun, stretched, and hardened to produce fibers suitable for textiles and for other purposes.

508. STABILIZATION OF VISCOUS DISPERSIONS OF COTTONSEED PROTEINS. Arthur, J. C., Jr. U. S. Pat. No. 2,531,383. November 28, 1950.

A process of stabilizing the viscosity of dispersions of cottonseed proteins comprising: forming a mixture of the proteins, water, a reducing sugar, (in the proportions of from 88 to 220 parts water and 0.45 to 4.5 parts reducing sugar per 22 parts of protein) and an alkali.

262. VEGETABLE PROTEIN HYDRATES. Burnett, R. S.; and Roberts, E. J. U. S. Pat. No. 2,421,113. May 27, 1947.

A process for preparing fluid, comparatively stable, and relatively clear vegetable protein hydrates having a pH of about 7. These hydrates are homogeneous solutions of water in proteins, in contrast to dispersions, which consist of heterogeneous mixtures. The hydrates are prepared by forming a mixture of water and protein in which the quantity of water is enough to completely hydrate the protein, but is not more than the amount that the protein molecules will bind, i. e., about 50 percent by weight.

SPECIAL INVESTIGATIONS - GOSSYPOL AND OTHER PIGMENTS

3100. THE OXIDATION OF GOSSYPOL. II. FORMATION OF GOSSYPOLONE WITH FERRIC CHLORIDE. Haas, R. H.; and Shirley, D. A. Contractor: Department of Chemistry, University of Tennessee, Knoxville, Tennessee. J. Org. Chem. 30, 4111-4113. 1965.

The oxidation of gossypol I with ferric chloride in acetic acid-acetone gave the 1, 4-binaphthoquinone II in good yield. Formation of II seemed due to complexation and stabilization of the o-hydroxyaldehyde moiety by the ferric chloride and steric blocking of the normal oxidative coupling reaction of phenols.

2090. NEW INFORMATION ON THE MORPHOLOGY OF THE GOSSYPOL PIGMENT GLAND OF COTTONSEED. Moore, A. T.; and Rollins, M. L. J. Am. Oil Chemists' Soc. 38: 156-60. 1961.

Exploratory electron microscope studies of the cottonseed pigment gland demonstrate a complex internal structure in which discrete particles ranging in size from 1 micron to less than 0.2 micron in diameter are held within a membranous mesh-like network. This structure is extremely sensitive to water, and it may be the rapid swelling of the network that results in the explosive release of pigment particles from the gland on exposure to moisture. Pigment particles are spherical, of a wide range of diameters, and exhibit no birefringence in the gland or when extruded. Calculations of specific surface, based on sizes of particles and density of purified gossypol (1.34), indicate surface area per gram of gossypol particles of the order of 8 square meters. Further work is indicated to determine details of the structure of the platelets, which constitute the wall of the gland, and to establish the relationship of gossypol and gossypurpurin to morphological features within the gland.

2064. SPONTANEOUS CONVERSION OF GOSSYPOL TO ANHYDROGOSSYPOL. Berardi, L. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 38 : 51-52. 1961.

An alkali-fast pigment is produced at room temperature when gossypol is dissolved in ethyl acetate. This pigment has been isolated from ethyl acetate as an orange crystalline material and identified as anhydrogossypol. The identity was established by identity of the infrared spectra with that of authentic anhydrogossypol; by elementary composition; by mixture-melting point behavior with authentic anhydrogossypol; and by the identity of the aniline derivatives produced from the orange crystalline product from ethyl acetate and authentic anhydrogossypol.

2061. COTTONSEED OIL PIGMENTS: FRACTIONATION BY MEANS OF MOLECULAR SIEVES, COUNTERCURRENT DISTRIBUTION, AND LOW-TEMPERATURE CRYSTALLIZATION. Verburg, G. B.; McCall, E. R.; O'Connor, R. T.; and Dollear, F. G. J. Am. Oil Chemists' Soc. 38: 33-39. 1961.

Crude cottonseed oils were shown to have spectra of two types, those exhibiting a single maximum at about 360 mμ and those exhibiting double maxima at about 375 and 400 mμ . Gossypol and cottonseed oil pigments that absorb below about 375 mμ are trapped by molecular sieve No. 13X, appear in the hydrophilic phase in countercurrent distribution of the oil between 95 percent ethanol and iso-octane, and are found in the liquid fraction on crystallization of oil from acetone at - 63° C. Those pigments not admitted by molecular sieve No. 13X are found in the hydrophobic phase in countercurrent distribution and in the solid fraction from low-temperature crystallization. Only countercurrent distribution achieved separation of both types of pigments from the triglycerides. Insufficient quantities of pigments were isolated for any extensive characterization. Use of large-scale apparatus should make it possible to separate the pigments for characterization and evaluation of their relation to the refining properties of oils.

1991. SOME NEW REACTIONS AND REACTION PRODUCTS OF APOGOSSYPOL AND DESAPOGOSSYPOL. Shirley, D. A.; and Alley, P. W. J. Org. Chem. 25 : 1391-94. 1960.

The previously unreported desapogossypol was prepared by demethylation of the hexamethyl ether. Desapogossypol was converted to the hexa-acetate, desapogossypolone tetra-acetate, and to hydrodesapogossypolone octa-acetate. The hexa-allyl ether of apogossypol has been prepared and carried through a Claisen rearrangement that involved two ortho- and two para-rearrangements in each molecule. Epoxidation of apogossypolone tetramethyl ether and desapogossypolone tetramethyl ether gave in both cases the corresponding 2, 3, 2', 3'-diepoxy derivatives.

1964. GOSSYPOL ACETIC ACID AS A REFERENCE STANDARD IN THE DETERMINATION OF GOSSYPOL. Hoffpauir, C. L.; Harris, J. A.; and Hughes, J. P. J. Assoc. Offic. Agr. Chemists 43 : 329-31. 1960.

Methods for the determination of gossypol in various cottonseed products, such as oil, meals, and feeds, require the use of pure gossypol as a reference standard. Because of its very reactive nature, pure gossypol suffers significant degradation upon extended storage, even under the best of conditions. The use of gossypol acetic acid instead of gossypol as a reference standard is suggested. Gossypol acetic acid, a 1:1 molar addition compound of gossypol and acetic acid, is more readily prepared and more easily purified than gossypol, and is somewhat more stable. It has a molecular weight of 578. 5, as compared with 518. 54 for gossypol, and hence contains 89. 62 percent actual gossypol. Accordingly, 27. 9 mg. of pure gossypol in the preparation of the reference standard solution for the determination of gossypol.

1893. SOME NEW ANIL DERIVATIVES OF GOSSYPOL. Alley, P. W.; and Shirley, D. A. J. Org. Chem. 24 : 1534-36. 1959.

The scope of the reaction of Schiff base (anil) formation between gossypol and primary amines has been investigated. Seventeen primary amines of widely varying types were reacted with gossypol and the anil derivates isolated.

1879. RECOVERY OF GOSSYPOL FROM COTTONSEED GUMS. Pons, W. A., Jr.; Pominski, Joseph; King, W. H.; Harris, J. A.; and Hopper, T. H. J. Am. Oil Chemists' Soc. 36 : 328-32. 1959.

A process has been developed for the isolation of pure gossypol from the gums obtained by water-washings of crude hexane-extracted cottonseed oil. Gums are heated with methyl ethyl

ketone containing phosphoric acid to cleave gossypol-phosphatide reaction products and are cooled to separate the ketone phase containing the gossypol from a phosphatide-water phase. After concentration by distillation, gossypol is isolated from the methyl ethyl ketone concentrate by addition of glacial acetic acid to form the acetic-acid addition compounds of 92 to 94 percent purity. Two recrystallizations as the acetic-acid complex produce gossypol acetic acid of 99 percent purity. The purified product can be dissociated by solution in dilute sodium carbonate, from which pure gossypol is recovered by acidification with mineral acid. Yield data from pilot-plant experiments indicated that about 47 percent of the gossypol in gums is recovered as crude gossypol acetic acid. Depending on the degree of purification of the crude product, the overall yield of purified gossypol from gums will range from 41 percent for a product of 98 percent purity to 36 percent for a gossypol of 99 percent purity.

1876. PRODUCTION OF GOSSYPOL FROM COTTONSEED GUMS. PRELIMINARY COST STUDY. Koltun, S. P.; Decossas, K. M.; Pominski, Joseph; Pons, W. A., Jr.; and Patton, E. L. J. Am. Oil Chemists' Soc. 36: 349-52. 1959.

On the basis of operations of a hypothetical gossypol-extraction plant, it is estimated that crude gossypol-acetic acid can be produced at a cost of $2.64 per pound at an annual production of 113,500 lb.; that pure gossypol-acetic acid can be produced at a cost of $3.80 per pound at an annual production of 100,200 lb.; and that pure gossypol will cost $5.55 per pound at an annual production of 81,000 lb. By marketing the phosphatides instead of returning them to the gums, the cost of producing pure gossypol will be $3.91 per pound at an annual production of 81,000 lbs. These costs were estimated by assuming that Phase I of the process would be accomplished simultaneously with oil mill-extraction operations, and Phase II and III during remainder of the season. It is apparent that gossypol or gossypol-acetic acid produced by this process is not inexpensive and would probably have to be used for specialized purposes, such as pharmaceuticals, where the cost would not be prohibitive.

1868. KINETIC STUDY OF GOSSYPOL FIXATION IN COTTONSEED OIL. Pons, W. A., Jr.; Berardi, L. C.; and Frampton, V. L. J. Am. Oil Chemists' Soc. 36: 337-39. 1959.

It was shown in experiments carried out under anaerobic conditions that the fixation of gossypol in cottonseed oil is a reaction of the second order with respect to gossypol. In other words, the rate of fixation is proportional to the square of the gossypol concentration in the oil. The rate of fixation depends on the temperature and increases 22-fold with an increase in temperature from 40° to 80° C.

1777. REPORT ON GOSSYPOL IN FEEDS: COLLABORATIVE STUDY OF METHODS FOR FREE AND TOTAL GOSSYPOL. Hoffpauir, C. L. J. Assoc. Offic. Agr. Chemists 42: 224-25. 1959.

A procedure for the determination of free and total gossypol in mixed feeds containing cottonseed meal was described last year. In this procedure free gossypol is extracted with a mixed isopropyl alcohol-hexane-water solvent containing a gossypol complexing agent, 3-amino-1-propanol, which prevents interference by feed constituents and stabilizes gossypol during the extraction. The same complexing agent dissolved in dimethylformamide is utilized for a rapid extraction of total gossypol from the feed. The complexed gossypol in the extracts reacts with aniline to completely convert the gossypol to dianilinogossypol; this reaction is the basis of the colorimetric determination. The method has been studied collaboratively in accordance with the previous recommendations. The collaborative results appear to offer considerable promise for the procedure.

1775. REACTION OF GOSSYPOL WITH FREE EPSILON-AMINO GROUPS OF LYSINE IN PROTEINS. Conkerton, E. J.; and Frampton, V. L. Arch. Biochem. Biophys. 81: 130-34. 1959.

The number of epsilon-amino groups of lysine in proteins that are free to react with 2, 4-dinitrofluorobenzene is reduced when the proteins are exposed to reaction with gossypol. The reduc-

tion in reactive epsilon-amino groups of lysine is increased when the pH of the reaction mixture is increased. A procedure for estimation of the epsilon-amino groups is described.

1677. BINDING OF GOSSYPOL UNDER CONDITIONS OF COMPLETE RUPTURE OF THE PIGMENT GLANDS. King, W. H.; Frampton, V. L.; and Altschul, A. M. J. Am. Oil Chemists' Soc. 35 : 358-60. 1958.

The free gossypol of cooked cottonseed meats is composed of gossypol and of gossypol-like pigments, which are soluble in 70 percent aqueous acetone. The gossypol-like pigments may account for 30 to 100 percent of the free gossypol as determined by the A. O. C. S. method. Thorough comminution followed by prolonged cooking results in reduction of all the pigments in cottonseed meats. Free gossypol is not completely eliminated, but the gossypol level, as determined by the benzene transfer method, may be reduced to zero. Gossypol-like pigments are converted into gossypol by the action of hot aqueous hydrochloric acid. Good agreement is observed between the data for total gossypol as determined through the use of the A. O. C. S. oxalic acid method and through the use of hot aniline.

1613. 3-AMINO-1-PROPANOL AS A COMPLEXING AGENT IN THE DETERMINATION OF TOTAL GOSSYPOL. Pons, W. A.; Pittman, R. A.; and Hoffpauir, C. L. J. Am. Oil Chemists' Soc. 35 : 93-97. 1958.

A method is proposed for the determination of total gossypol in cottonseed meals, crude oils, and soapstocks based on a rapid extraction of gossypol by neutralized 3-amino-1-propanol in dimethylformamide to form a stable complex, followed by colorimetric analysis of an aliquot of the extract by means of an aniline reaction. A determination can be completed in about 2 hours and with minor modification in 1 hour compared to about 7 hours for current methods. Results obtained by the proposed procedure on meals, oils, and soapstocks are in essential agreement with those found by use of other accepted methods. Desirable features, such as stability of reagents and extracts and a high degree of reproducibility, suggest that the procedure will satisfy the requirements for a rapid and simplified method for the analysis of all cottonseed products for total gossypol.

1539. THE PIGMENTS OF CRUDE COTTONSEED OILS. II. NITROGEN-CONTAINING PIGMENTS DERIVED FROM GOSSYPOL. Dechary, J. M. J. Am. Oil Chemists' Soc. 34 : 597-600. 1957.

Dark crude cottonseed oil has been shown to contain a mixture of ninhydrin-sensitive substances combined with gossypol. Phenylalanine, aspartic acid, and glycine have been obtained by acid hydrolysis of the addition compound, followed by paper chromatography of the hydrolysate. The presence of other amino acids was indicated, but their identification could not be established with certainty; proline and hydroxy-proline were apparently absent. The possibility that part of the color of dark crude oils arises from gossypol-alpha-amino acid interaction is discussed.

1533. INCORPORATION OF GOSSYPOL INTO EGGS OF HENS FED GOSSYPOL SCHIFF BASES. Dechary, J. M.; Wakabayashi, G.; and Grau, C. R. J. Am. Oil Chemists' Soc. 34 : 548-49. 1957.

Eggs from hens fed gossypol Schiff bases derived from various aromatic and aliphatic amines were examined spectrophotometrically for gossypol-cephalin. Aniline, p-aminobenzoic acid, dehydroabietylamine, and cyclohexylamine were not effective gossypol binders when the Schiff base was fed at the 0. 5 percent equivalent gossypol level in the diet, whereas the long-chain aliphatic amines were very effective. Hens produced normal eggs when fed the 0. 5 percent level of gossypol Schiff bases derived from these amines.

1528. DETERMINATION OF FREE AND TOTAL GOSSYPOL IN MIXED FEEDS CONTAINING COTTONSEED MEALS. Pons, W. A., Jr.; and Hoffpauir, C. L. J. Assoc. Offic. Agr. Chemists 40 : 1068. 1957.

Analytical methods are described for the determination of free and total gossypol in mixed feeds containing cottonseed meal. Free gossypol is extracted with a mixed isopropyl alcohol-hexane-water solvent containing a gossypol complexing agent, 3-amino-1-propanol, which prevents interference by feed constituents and stabilized gossypol during the extraction. The same complexing agent dissolved in dimethylformamide is utilized for a rapid extraction of total gossypol from the feed. Reaction of the complexed gossypol in the extracts with aniline results in complete conversion of the gossypol to dianilinogossypol, and serves as the basis for the colorimetric determination. The procedures are not affected by ingredients normally present in feeds, and allow estimation of free and total gossypol in the range normally expected in commercial feed.

1459. THE STRUCTURE AND REACTIONS OF GOSSYPOL. V. METHYLAPOGOSSYPOL HEXAMETHYL ETHER AND 2,3-DIMETHOXY-4-ISOPROPYL-5-ALLYLTOLUENE. Shirley, D. A.; Brody, S. S.; and Sheehan, W. C. J. Org. Chem. 22 : 495-97. 1957.

Reduction of gossypol with lithium aluminum hydride followed by methylation yields a product designated as methylapogossypol hexamethyl ether, in which the aldehyde groups of the gossypol molecule have been reduced to methyl groups and the hydroxyl groups methylated. Work directed toward the synthesis of methylapogossypol hexamethyl ether has resulted in the seven-step preparation of a key intermediate, 2,3-dimethoxy-4-isopropyl-5-allyltoluene.

1458. THE STRUCTURE AND REACTIONS OF GOSSYPOL. IV. THE SYNTHESIS OF DESAPOGOSSYPOL HEXAMETHYL ETHER. Shirley, D. A.; and Dean, W. L. J. Am. Chem. Soc. 79 : 1205. 1957.

1, 1', 6, 6', 7, 7'-Hexamethoxy-3, 3'-dimethyl-2, 2'-binaphthyl has been synthesized and found to be identical with desapogossypol hexamethyl ether, a degradation product of gossypol.

1456. REPORT ON GOSSYPOL IN FEEDS. INVESTIGATION OF EXTRACTION CONDITIONS. Hoffpauir, C. L.; and Pons, W. A., Jr. J. Assoc. Offic. Agr. Chemists 40 : 363-69. 1957.

Recovery of free gossypol from cottonseed meal in admixture with commercial mixed chick feed was incomplete when methyl ethyl ketone containing 10 percent water was used in either a single or two-step equilibrium extraction. Incorporation of metal chelating agents in the solvent did not significantly improve the recoveries. Attempts to obtain complete extraction of free gossypol by use of gossypol complexing agents were also unsuccessful. Similar results were obtained when pure gossypol was added to the commercial chick feed. The results suggest that continuous extraction (Soxhlet or Butt) will probably be required in order to completely extract free gossypol from mixed feed containing cottonseed meal.

1309. THE STRUCTURE AND REACTIONS OF GOSSYPOL. III. ALIPHATIC ANIL DERIVATIVES OF GOSSYPOL. Shirley, D. A.; and Sheehan, W. C. J. Org. Chem. 21 : 251. 1956.

Stable anils and a variety of primary aliphatic amines were prepared from gossypol by heating equivalent quantities of the reactants in isopropyl alcohol. Gossypol and dodecylamine produced didodecylaminogossypol in 95 percent yield. Properties of several aliphatic anil derivatives of gossypol are tabulated.

1308. SUBSTITUTED ARYLIMINO DERIVATIVES OF GOSSYPOL. Dechary, J. M.; and Brown, L. E. J. Am. Oil Chemists' Soc. 33 : 76-78. 1956.

Seventeen substituted arylimino derivatives of gossypol have been prepared and characterized. Fifteen of these are new compounds. O-Nitroaniline gave both a mono- and a disubstituted derivative with gossypol whereas p,p'-methylenedianiline gave only a mono-substituted derivative. During experiments designed to prevent color reversion in stored, crude cottonseed oils by treatment with various aromatic amines, only p-aminobenzoic acid formed an oil-insoluble derivative with the gossypol in the oils.

1306. REPORT ON GOSSYPOL IN FEEDS--INTERFERENCE OF FEED CONSTITUENTS IN THE FREE GOSSYPOL. Hoffpauir, C. L.; and Pons, W. A., Jr. J. Assoc. Offic. Agr. Chemists 39 : 300-305. 1956.

The recovery of free gossypol from cottonseed meal added to typical chick mixed feeds has been investigated. Extraction of gossypol with 70 percent acetone was unsatisfactory because of interference by soluble constituents of the feed. Use of 90 percent methyl ethyl ketone in the presence of chelating agents to eliminate interference by metal ions, resulted in substantially higher recoveries, but even the highest values obtained represent less than 90 percent of the gossypol added.

1304. IMPROVED METHOD FOR DETERMINING GOSSYPOL IN CRUDE COTTONSEED OILS. Pons, W. A., Jr.; Mitcham, Donald; O'Connor, R. T.; and Stansbury, M. F. J. Am. Oil Chemists' Soc. 33 : 324-30. 1956.

The p-anisidine method for the determination of gossypol in crude cottonseed oils has been reinvestigated and modified to make it applicable to all crude oils obtained by the newer methods of processing cottonseed. The modifications included a change in the composition of the solvent, a higher reaction temperature, and the use of a more concentrated p-anisidine reagent. The modified method was found satisfactory where different colorimeters and spectrophotometers were used for measuring the color developed. Comparison of aniline and p-anisidine as reagents for the analysis of gossypol pigments showed that the presence of modified gossypol in some crude oils resulted in an over-correction for background absorption and led to significant errors when aniline was used as the color development agent.

1301. CONVERSION OF DIANILINOGOSSYPOL TO PURE GOSSYPOL. King, W. H.; and Thurber, F. H. J. Am. Oil Chemists' Soc. 33 : 169-71. 1956.

A procedure for isolation of gossypol (in 86 percent yield) from dianilinogossypol, together with a procedure for the purification of gossypol, is described. Dianilinogossypol is hydrolyzed in alcoholic potassium hydroxide, and the hydrolysis mixture is rapidly cooled and acidified, whereupon the gossypol is precipitated and the aniline remains in solution as the salt. Directions are given for the separation of gossypol from unhydrolyzed dianilinogossypol through partition between aqueous alkali and chloroform.

1232. STRUCTURE AND REACTIONS OF GOSSYPOL. II. THE SYNTHESIS OF 1, 6, 7-TRIMETHOXY-3-METHYLNAPHTHALENE, A PROTOTYPE OF DESAPOGOSSYPOL HEXAMETHYL ETHER. Shirley, D. A.; and Dean, W. L. J. Am. Chemical Soc. 77 : 6077-79. 1955.

An attempt was made to synthesize a compound more closely related to gossypol than desapogossypolone hexamethyl ether, one that would provide additional information on what is generally assumed to be the molecular structure of gossypol. 3-Methyl-6, 7-dimethoxy-1-tetralone was converted, through several reactions, to obtain 1, 6, 7-trimethoxy-3-methylnaphthalene. At this point it was found that the ultraviolet absorption spectrum of this differed appreciably from that of desapogossypol hexamethyl ether. This difference in the absorption spectra was interpreted as indicating an attachment of the two naphthol rings through the 3, 3'-positions rather than through the 2, 2'-positions.

1227. REDUCTION OF GOSSYPOL WITH LITHIUM ALUMINUM HYDRIDE. Shirley, D. A.; and Sheehan, W. C. J. Am. Chemical Soc. 77 : 4606-08. 1955.

The reduction of gossypol with lithium aluminum hydride followed by acetylation yields a product designated as methylapogossypol hexa-acetate. The aldehyde groups of gossypol have been reduced by methyl groups and all hydroxyl groups acetylated. Oxidation of methylapogossypol hexa-acetate yields methylapogossypolone tetra-acetate. Reductive acetylation of this latter product leads to methylapohydrogossypolone octa-acetate.

1222. INTERACTION OF GOSSYPOL WITH THE FERROUS ION. Jonassen, J. B.; and Demint, R. J. J. Am. Oil Chemists' Soc. 32 : 424-26. 1955.

Several investigators have used iron salts to inactivate gossypol, and the object of this investigation was to determine the combining ratios for the reaction between gossypol and the ferrous ion. The preparation of a solution of sodium gossypolate in aqueous acetone has been described. Indications of a 1:1 mole ratio combination of the gossypolate and ferrous ions were established through conductometric and potentiometric studies in the presence of either chloride or perchlorate anion. Application of the method of continuous variations to differences in the absorption spectra of the two species confirmed the 1:1 mole ratio of ferrous ion to gossypol. This finding is supported by analytical data. Values of pK for the dissociation constant of ferrous gossypolate, calculated from spectrophotometric measurement, averaged 7. 3.

1220. FREE GOSSYPOL AND PROTEIN SOLUBILITY. Stansbury, M. F. Oil Mill Gaz. 60 (4) : 29-30. 1955.

In reports on the progress of research to improve the nutritive value and utility of cottonseed meal, constant reference has been made to analytical methods for determining free gossypol content and protein solubility. This communication discusses these two analytical procedures, some of the precautions to be observed in their use, and the precision which can be expected. The precision of analysis of 184 conventional cottonseed meals for free gossypol at the Southern Laboratory is compared with the results of check-sample testing by the Smalley Committee of the American Oil Chemists' Society. Results of check-sample testing of the protein solubility method are also summarized. Free gossypol contents and protein solubilities of hydraulic-press, normal screw-press, low-temperature screw-press, prepress solvent-extraction, and filtration-extraction cottonseed meals are summarized.

1163. REPORT ON GOSSYPOL IN FEEDS. EFFECT OF VARIATION IN SAMPLE WEIGHTS AND ALIQUOTS ON DETERMINATION OF FREE GOSSYPOL. Hoffpauir, C. L., Associate Referee; and Pons, W. A., Jr. J. Assoc. Offic. Agr. Chem. 38 : 234-38. 1955.

The application of accepted methods for the determination of free gossypol in cottonseed meal to the analysis of mixed feeds requires the use of increased sample weights or increased aliquots or both. Modification of the color development to use p-anisidine at 80° C. permits the use of larger aliquots, but the sample size is limited to one gram or less, as use of larger samples results in incomplete extraction of the free gossypol.

1162. RELATIONSHIP BETWEEN DISCOLORATIONS IN EGGS AND DIETARY FREE GOSSYPOL SUPPLIED BY DIFFERENT COTTONSEED PRODUCTS. Heywang, B. W.; Bird, H. R.; and Altschul, A. M. Poultry Sci. 34 : 81-90. 1955.

Pullets were fed all-mash diets containing free gossypol at levels of 0. 001, 0. 002, 0. 003, 0. 004, 0. 005, and 0. 008 percent. It was found that, in general, the lower the percentage of free gossypol, the less discoloration appeared in the eggs; with gossypol content as high as 0. 008 percent, probability of discoloration was great. When eggs were placed in cold storage, discoloration increased with the length of the storage period, both in extent and number of eggs affected.

1161. EFFECT OF TIME AND TEMPERATURE OF STORAGE ON THE FREE AND TOTAL GOSSYPOL CONTENT OF COTTONSEED MEALS AND OF MIXED DIETS. Kupperman, R. P.; and Karon, M. L. J. Am. Oil Chemists' Soc. 32 : 54-57. 1955.

In different types of cottonseed products, and in cottonseed products to which gossypol is added, there is a loss or disappearance of gossypol upon storage, as determined analytically. The rate of disappearance of gossypol varied for the different types of cottonseed and increased at higher temperatures and over the longer periods of storage. When gossypol is incorporated into different types of animal diet materials, there is a loss or inactivation of some portion of the added gossypol as determined analytically immediately after preparation of the gossypol-diet mixtures, and a further additional loss or inactivation upon storage.

1160. DETERMINATION OF FREE GOSSYPOL IN CHEMICALLY TREATED COTTONSEED MEALS CONTAINING DIANILINOGOSSYPOL. Pons, W. A., Jr; and Hoffpauir, C. L. J. Am. Oil Chemists' Soc. 32 : 295-300. 1955.

A method is proposed for the determination of free gossypol in chemically treated cottonseed meals containing dianilinogossypol. The procedure consists of the extraction of free gossypol with 70 percent acetone containing 0. 5 aniline per liter, followed by colorimetric determination by means of reaction with aniline at 100° C. Changes in the optical density of 70 percent acetone extracts of chemically treated meals, due to slow hydrolysis of dianilinogossypol, were prevented by addition of trace quantitles of aniline to the aqueous acetone for extraction. The proposed procedure gave constant values for free gossypol over a period of several hours after extraction. When aqueous acetone without aniline was used, free gossypol values increased with the time interval between extraction and analysis. Extrapolation of these values to zero time gave results in good agreement with those obtained by use of aqueous acetone containing aniline.

1100. THE PIGMENTS OF CRUDE COTTONSEED OILS. I. THE INHIBITION OF COLOR REVERSION IN CRUDE COTTONSEED OILS. Dechary, J. M.; Kupperman, R. P.; Thurber, F. H.; and O'Connor, R. T. J. Am. Oil Chemists' Soc. 31 : 420-24. 1954.

Treatment of freshly prepared crude cottonseed oils with p-aminobenzoic acid and subsequent removal of the di-p-carboxyanilinogossypol formed makes it possible to store the oils at 37 to 38° C. for 30 days without incurring any adverse changes in the refining and bleaching properties of the oils. Refining loss of the crude oil is considerably decreased, and the stability of the bleached oil is not affected by the treatment. Spectrophotometric studies show that the p-aminobenzoic acid removes almost completely the gossypol-like pigments that are present in the crude oils and yields oils having the characteristic carotenoid spectrum.

1011. THE INFRARED SPECTRA OF GOSSYPOL. O'Connor, R. T.; Von Der Haar, Patricia; DuPre', E. F.; Brown, L. E.; and Pominski, C. H. J. Am. Chemical Soc. 76 :2368-73. 1954.

Gossypol creates problems in processing cottonseed because it lowers quality of the meal and, when extracted with oil, is responsible for color reversion. Spectrophotometric studies to correlate infrared spectra with chemical groupings in the molecules of gossypol and its derivatives have provided a basis for following by infrared absorption the changes occurring in these materials. Infrared spectra of chloroform solutions of gossypol, of 13 derivatives and degradation products of gossypol, and 18 "model" compounds containing groupings present in the gossypol molecule have been obtained in the region from 2 to 12 mμ. The data confirm that gossypol is an aromatic and phenolic compound; that a carbonyl ortho to a hydroxyl group is present; that in some derivatives there are no carbonyl groups; and that in others carbonyl groups are attached directly to an alicyclic grouping.

1009. THE ANTIOXIDANT AND ANTIPOLYMERIZATION PROPERTIES OF GOSSYPOL, DIANILINOGOSSYPOL, AND RELATED MATERIALS. Bickford, W. G.; Pack, F. C.; Castillon, L. E.; and Mack, C. H. J. Am. Oil Chemists' Soc. 31 : 91-93. 1954.

Because of the potential availability of about 40,000 tons of gossypol annually this and related materials have been evaluated for their antioxidant and antipolymerization properties. Gossypol was superior as an antioxidant; and dianilinogossypol was the most effective as an antipolymerization agent. Antioxidant substrates were cottonseed oil and lard. Styrene, vinyl acetate, and methyl acrylate were the vinyl-type monomers chosen for antipolymerization test. Monomers protected with gossypol or related materials are highly colored--a fact limiting their outlets. The toxicological properties of certain materials tested are undetermined; however, the concentrations at which gossypol is effective are below reported deleterious physiological levels.

961. THE PIGMENTATION OF "RED" COTTONSEED OIL. Pominski, C. H.; Von Der Haar, Patricia; O'Connor, R. T.; and Coleman, W. T. J. Am. Oil Chemists' Soc. 30 : 365-68. 1953.

Information was obtained concerning the pigments responsible for red color in cottonseed oils. A crude screw-pressed cottonseed oil of unusually high red color was treated with dilute hydrochloric acid before alkali refining. For comparison, the same acid treatment was applied to oil expressed from corresponding seed in the pilot-plant hydraulic press without the application of heat. A dark red pigment resembling an anthocyanin was isolated. It seems likely that anthocyanins, their precursors, or their decomposition products contribute to the dark color of "red" screw-pressed oils which are difficult to refine and bleach.

940. GOSSYPOL IN COTTONSEED. INFLUENCE OF VARIETY OF COTTONSEED AND ENVIRONMENT. Pons, W. A., Jr.; Hoffpauir, C. L.; and Hopper, T. H. J. Agr. Food Chem. 1: 1115-18. 1953.

The first systematic investigation of the influence of variety of cottonseed and environment on the gossypol content of kernels is reported. Data are reported for the gossypol content of kernels from the seed of 8 varieties of cotton grown at 13 locations during 3 years. Values ranged from 0.39 to 1.70 percent gossypol in the moisture-free kernels. Analysis of variance showed that both variety and environment significantly influenced these values. Standard deviations of the means suggested that these effects are about equal in their influence.

938. REVIEW OF THE PROPERTIES OF GOSSYPOL AND METHODS OF ITS ESTIMATION. Hoffpauir, C. L.; and Pons, W. A., Jr. J. Offic. Agr. Chemists 36: 1108-18. 1953.

This article reviews 47 references giving information on the physical and chemical properties of gossypol and analytical methods for its determination. The article is illustrated by four figures, showing (1) tautomeric forms of gossypol, (2) ultraviolet absorption spectra of gossypol in cyclohexane, (3) ultraviolet absorption spectra of cottonseed pigments in chloroform, and (4) absorption spectra of 70 percent aqueous acetone extracts of various cottonseed.

868. AN IMPROVED PROCEDURE FOR THE PURIFICATION OF GOSSYPOL. King, W. H.; and Thurber, F. H. J. Am. Oil Chemists' Soc. 30: 70-74. 1953.

Methods of dissociating the gossypol-acetic acid complex from the butanone extract of defatted cottonseed flakes and methods of recrystallizing gossypol were studied. The study led to the development of an improved rapid procedure for the purification of gossypol. The degree of purity of the gossypol prepared by the improved procedure was determined by a number of tests, which are described. Spectral and titration curves for pure gossypol are presented, and standards of purity are suggested. The molecular weight of gossypol and gossypol-acetic acid prepared by the procedures described is approximately 518 and 578, respectively.

865. PREPARATION OF COMBINATION PRODUCTS OF GOSSYPOL WITH GLYCINE AND DEXTROSE AND THEIR TOXICITY TOWARD MICE. Castillon, L. E.; Karon, M. L.; Altschul, A. M.; and Martin, F. N. Arch. Biochem. and Biophys. 44: 181-88. 1953.

Combination products of gossypol with glycine and with dextrose have been prepared. Acute oral toxicity investigations indicate that the freshly prepared gossypol-dextrose combination product is more toxic than isolated gossypol and as toxic as isolated pigment glands. The gossypol-glycine combination product is nontoxic when administered in dosages as large as 12.0 g./kg. body weight of mice. Purified gossypol-glycine has been isolated and found to be a water-soluble, nitrogen-containing compound. It differs from gossypol in many of its properties.

814. REMOVAL OF GOSSYPOL FROM COTTONSEED BY SOLVENT-EXTRACTION PROCEDURES. Dechary, J. M.; Kupperman, R. P.; Thurber, F. H.; and Altschul, A. M. J. Am. Oil Chemists' Soc. 29: 339-41. 1952.

The relative efficiencies of organic, polar solvents and of solvent-water pairs for use in the extraction of gossypol and related compounds from cottonseed flakes were determined in a specially devised glass laboratory extractor. A butanone-water pair containing 10 percent of water by volume was the most effective, and chlorine-substituted hydrocarbons were the least effective. Flakes extracted at 26° C. contained 0.108 percent free gossypol and those extracted at 71°, 0.054 percent. The decrease may be due, in part, to the reaction of gossypol with the protein to form bound gossypol.

751. THE LOSS OR INACTIVATION OF PURE GOSSYPOL IN A MIXED DIET. Heywang, B. W.; Bird, H. R.; and Kupperman, R. P. Poultry Sci. 31: 35-39. 1952.

Most of the gossypol mixed with a diet (for poultry) at the 0.012, 0.024, and 0.036 percent levels was undetectable by chemical analysis after the mixtures had stood for several days. To learn whether the gossypol had been destroyed or rendered inactive for chickens, a 42-day experiment was conducted with four groups of 10 White Leghorn pullets each--selected because of the high hatchability of their eggs when all had been fed the basal diet containing no gossypol. During the 42 days, the basal diet contained pure gossypol mixed at the 0.0-, 0.012-, 0.024-, or 0.036-percent levels. The mixed diets were fed on the 6th and 7th days after mixing. There was no decrease in hatchability with gossypol at the 0.012- and 0.024-percent levels, and only a slight decrease at the 0.036- percent level. There was no adverse effect on whole egg weight at any of the three levels.

722. PIGMENTS OF COTTONSEED. V. METHYLATION OF GOSSYPURPURIN. Von Der Haar, Patricia; and Pominski, C. H. J. Org. Chem. 17: 177-80. 1952.

The methylation of gossypol gave the completely methylated ether, hexamethoxygossypol. Methylation of gossypurpurin produced an ether whose methoxyl value indicated the presence of four methoxyl groups and which resisted further methylation. Molecular weight determinations on the two ethers showed the molecular size of the gossypurpurin methyl ether to be approximately twice that of hexamethoxygossypol. A tentative molecular formula for the gossypurpurin methyl ether was proposed on the basis of its elementary analysis, methoxyl value, and molecular weight.

658. PIGMENTS OF COTTONSEED. IV. GOSSYPURPURIN. A PURPLE PIGMENT RELATED TO GOSSYPOL. Pominski, C. H.; Miller, C. B.; Von Der Haar, Patricia; O'Connor, R. T.; Castillon, L. E.; and Brown, L. E. J. Am. Oil Chemists' Soc. 28: 472-75. 1951.

The properties of gossypurpurin prepared from gossypol via diaminogossypol were compared with those of gossypurpurin isolated from pigment glands. A tentative molecular formula for synthetic gossypurpurin, $C_{30}H_{32}O_7N$, has been proposed on the basis of its elementary composition. The native pigment could not be obtained in the same degree of purity as the synthetic product, and the analytical data could therefore not be brought into exact agreement for the two

products. However, solutions of both pigments in chloroform exhibited almost identical absorption spectra and had identical antimony trichloride tests. Qualitative reactions seem to indicate that the functional groups of both the native and the synthetic gossypurpurin are identical, and the ready conversion of both products to gossypol upon contact with acid seems to indicate that their basic structures are similar.

657. REACTIONS OF SOME GOSSYPOL-LIKE PIGMENTS WITH ANILINE AND p-ANISIDINE. Pominski, C. H.; and Von Der Haar, Patricia. J. Am. Oil Chemists' Soc. 28: 444-46. 1951.

Gossypurpurin and diaminogossypol have been shown to react with aniline to give the same reaction product as does gossypol. These pigments also react with p-anisidine to give products whose absorption spectra exhibit maxima in the same positions as that of the reaction product of gossypol and p-anisidine.

656. STORAGE OF GOSSYPOL. Pominski, C. H.; Castillon, L. E.; Von Der Haar, Patricia; Brown, L. E.; and Damare, Hilton. J. Am. Oil Chemists' Soc. 28: 352-53. 1951.

Pure gossypol was stored for 18 months at temperatures of 3° and 23 to 28° C. in the presence or absence of light and air. On the basis of combustion data, absorption spectra, antimony trichloride tests, and melting points of the original and stored samples, it can be concluded that gossypol can be stored without deterioration for at least 18 months at temperatures from 3° to 23 to 28° C. in the absence of light, irrespective of the presence or absence of air.

589. REMOVING THE GLANDS FROM COTTONSEED. Pominski, C. H.; Castillon, L. E.; and Dechary, J. M. Yearbook Agr., U. S. Dept. Agr., 1950-51: 558-60.

Research on the nature and properties of the pigment glands of cottonseed at the Southern Regional Research Laboratory is discussed. The pigment glands are surrounded by a wall that is mechanically very strong and, although very susceptible to the action of water, very resistant to rupture in the presence of certain organic liquids. On the basis of a difference between the density of the pigment glands and that of the other seed tissue and the resistance of the pigment glands to rupture in certain liquids, a method has been devised for separating cottonseed into pigment glands, oil, and gland-free meal. The glands are the only parts of cottonseed that as yet have no commercial value. The method developed to remove the glands has made available sufficient quantities of pigment glands, as well as gland-free meal, for extensive investigation of their physical, chemical, and physiological properties.

525. DETERMINATION OF TOTAL GOSSYPOL PIGMENTS IN COTTONSEED OILS. Pons, W. A., Jr.; Hoffpauir, C. L.; and O'Connor, R. T. J. Am. Oil Chemists' Soc. 28: 8-12. 1951.

A method is proposed for the application of the p-anisidine-gossypol reaction to the analysis of cottonseed oils for total gossypol pigments. The oil is dissolved in a hexane-isopropanol solvent, and gossypol pigments are determined in an aliquot of the solution by means of the color developed with p-anisidine. The effect of the solvent and of the oil concentration on the color developed in the method has been investigated. The stability of gossypol in the solvent employed has been demonstrated, and data are presented on the recovery of gossypol added to cottonseed oils. Further spectrophotometric evidence indicated that a number of gossypol-like pigments are present in the crude oils and that they react with p-anisidine to give reaction products spectrophotometrically identical with that obtained for pure gossypol with the same reagent.

494. THE EFFECT OF PURE GOSSYPOL ON EGG HATCHABILITY AND WEIGHT. Heywang, B. W.; Bird, H. R.; and Altschul, A. M. Poultry Sci. 29: 916-20. 1950.

Ten White Leghorn pullets for each of four experimental groups for feeding tests were chosen for a high average of egg hatchability. One group was fed the diet with no gossypol and the others the diet in which pure gossypol was mixed at the .012, .024, and .036 percent levels. The mixed diets were fed only on the day of mixing and the following day. Gossypol had an appreciable adverse effect on egg hatchability and weight when it was mixed with the diet at the 0.024-percent level and a still greater adverse effect at the 0.036-percent level but did not affect either at the 0.012-percent level. Hatchability and egg weight returned to normal after gossypol feeding was stopped.

493. EFFECT OF FRACTIONATION AND TREATMENT ON THE ACUTE ORAL TOXICITY AND ON THE GOSSYPOL AND GOSSYPURPURIN CONTENT OF COTTONSEED PIGMENT GLANDS. Eagle, Edward; Hall, C. M.; Castillon, L. E.; and Miller, C. B. J. Am. Oil Chemists' Soc. 27: 300-303. 1950.

The acute oral toxicity in rats was determined for a number of cottonseed pigment gland preparations and for pure gossypol. No correlation was found between the toxicity of the various samples of glands and their extractable gossypol or gossypurpurin content. Fractions of glands were prepared and tested for toxicity. A water- and acetone-soluble fraction was more toxic than the original glands; but an acetone-insoluble residue has practically no toxicity. Dry heating had no apparent effect on the toxicity of the pigment glands, whereas heating in the presence of water markedly decreased their toxicity. Toxicity of pigment glands decreased with the length of time the original seed had been stored. There was no apparent change in toxicity when separated glands were stored except in those obtained from cottonseed which had first been defatted with hexane. The acute oral toxicity of pure gossypol in rats was less than that for preparations of glands which had extractable gossypol contents ranging from 24 to 90 percent. These results confirm a previous conclusion that the toxicity of cottonseed pigment glands is attributable either to some component or components of the glands other than, or in addition to gossypol, or to one which enhances the activity of gossypol.

491. PREPARATION OF WATER-SOLUBLE COMBINATION PRODUCTS OF GOSSYPOL AND THEIR TOXICITY TO AQUARIUM FISH. Castillon, L. E.; and Altschul, A. M. Soc. Expt. Biol. and Med. Proc. 74: 623-26. 1950.

The purpose of this investigation was to make combination products of gossypol with naturally occurring materials that would have physical properties different from those of the original material and to determine the effect of such changes on some physiological actions of gossypol. Gossypol was tested on aquarium goldfish, and sharp differences in physiological activity between the original gossypol and the combination products were obtained. Gossypol was combined with proteins, amino acids, and carbohydrates to produce materials that were soluble or dispersible in water or buffer solutions at pH 7.0 by mixing gossypol and the other material in aqueous solution at alkaline pH, neutralizing that mixture, and drying it by lyophilization. These water-soluble products were lethal to goldfish at concentrations as small as 1 part in 100,000 parts of water. An aqueous solution of gossypol peanut protein maintained its toxicity and its original gossypol content for at least 48 hours. Cottonseed pigment glands were shown to be less toxic than the gossypol combination complexes. Crystalline gossypol, when added to the aquarium bath, displayed no visible toxic effects on the goldfish.

472. DETERMINATION OF TOTAL GOSSYPOL PIGMENTS IN COTTONSEED MATERIALS. Pons, W. A., Jr.; Hoffpauir, C. L.; and O'Connor, R. T. J. Am. Oil Chemists' Soc. 27: 390-93. 1950.

A method for the determination of total gossypol pigments in cottonseed materials is described that involves an acid hydrolysis of the "bound" gossypol pigments in aqueous methyl ethylketone and the colorimetric determination of the gossypol pigments by the method described by Pons

and Guthrie for free gossypol pigments. The conditions for hydrolysis have been investigated for various types of cottonseed materials, and data are presented on the recovery of gossypol added to such materials. Spectrophotometric data indicate that the pigments in the hydrolyzates are gossypol and closely related pigments and that the conditions specified do not alter them.

423. DETERMINATION OF FREE GOSSYPOL IN COTTONSEED MATERIALS. Pons, W. A., Jr.; and Guthrie, J. D. J. Am. Oil Chemists' Soc. 26: 671-76. 1949. (Republished: Oil Mill Gaz. 54: 11, 13-18. 1950.)

A method for the determination of free gossypol in cottonseed materials is described. The gossypol is extracted with a measured volume of 70 percent aqueous acetone on a shaker for 1 hour, then is filtered. A colorimetric analysis of the filtrate is made by means of the reaction between gossypol and p-anisidine. The conditions for complete extraction of gossypol from various types of cottonseed materials were investigated, and the stability of gossypol in aqueous acetone was demonstrated. Data also are presented on the recovery of gossypol added to cottonseed materials.

389. GOSSYPOL AND GOSSYPURPURIN IN COTTONSEED OF DIFFERENT VARIETIES OF G. BARBADENSE AND G. HIRSUTUM, AND VARIATION OF THE PIGMENTS DURING STORAGE OF THE SEED. Boatner, C. H.; Castillon, L. E.; Hall, C. M.; and Neely, J. W. J. Am. Oil Chemists' Soc. 26: 19-25. 1949.

The initial contents of moisture, lipids, nitrogen, gossypol, and gossypurpurin were determined on seed from several varieties of cotton, after which the samples were stored under identical conditions and analyzed for gossypol and gossypurpurin at fixed intervals over a period of a year. Seed of the species G. barbadense contained more gossypol and very much more gossypurpurin than seed of the species G. hirsutum. Within the species G. barbadense, Sea Island seed contained more gossypol and less gossypurpurin than Egyptian seed. The content of gossypurpurin increased during storage of all of the seed, whereas that of gossypol varied in a number of different ways.

352. PREPARATION OF GOSSYPOL FROM COTTONSEED PIGMENT GLANDS. Castillon, L. E.; Hall, C. M.; and Boatner, C. H. J. Am. Oil Chemists' Soc. 25: 233-36. 1948.

Because the nature of gossypol, as yet difficult to obtain in pure form, suggests many possibilities for use as an antioxidant and in the pharmaceutical field, practical methods were sought for its extraction from cottonseed pigment glands and for its isolation from pigment gland extracts. The pigment glands used in the experiments were produced from whole cottonseed meal and from defatted cottonseed meal by the gland flotation process. Of the methods developed during the investigation, extraction with acetone followed by precipitation of gossypol acetic acid from the extract was found to be the preferable one for obtaining pure gossypol in good yields. Since this method is rapid and employs inexpensive and easily handled reagents, it is readily adaptable to the production of gossypol on a large scale. Precautions that must be taken in applying the method to large-scale production are discussed, together with methods for the purification and preservation of gossypol.

351. ACUTE ORAL TOXICITY OF GOSSYPOL AND COTTONSEED PIGMENT GLANDS FOR RATS, MICE, RABBITS, AND GUINEA PIGS. Castillon, L. E.; Hall, C. M.; and Boatner, C. H. Arch. of Biochemistry 18: 271-77. 1948.

The cottonseed glands added to diets of fasting rats, mice, guinea pigs, and rabbits proved highly toxic, causing death with large doses, whereas an equivalent amount of pure gossypol affected the animals very little. No correlation was apparent between the toxicity of different samples of pigment glands and their relative contents of either gossypol or gossypurpurin. These results add to the evidence that some component of cottonseed pigment glands other than, or in addition to, gossypol and gossypurpurin is responsible for their toxicity. Thermally, the

toxic material is relatively stable, but it is partly inactivated by the combined action of heat and water. It is not extracted from the glands by petroleum naphthas and tetrachloroethylene but is partly extracted by ethanol and is completely extracted by diethyl ether and acetone.

329. APPLICATION OF ANTIMONY TRICHLORIDE-SPECTROPHOTOMETRIC METHOD TO THE DETERMINATION OF GOSSYPOL IN COTTONSEED AND COTTONSEED PRODUCTS. Hall, C. M.; Castillon, L. E.; Guice, W. A.; and Boatner, C. H. J. Am. Oil Chemists' Soc. 25: 457-61. 1948.

A technique is described that permits application of the antimony trichloride-spectrophotometric method to the determination of gossypol in a variety of cottonseed products. Gossypol is determined by means of the following series of operations: (1) extraction of gossypol from cottonseed or cottonseed products by use of chloroform or aqueous ethanol; (2) isolation from the extracts by use of aqueous alkali; and (3) application of the antimony trichloride-spectrophotometric test. Data are presented to show the results obtained by application of this procedure to the determination of gossypol in pigment glands, raw cottonseed meats, cooked cottonseed meats, hydraulic- and screw-pressed meals, solvent-extracted meals, gland-free meals, and oils, both expressed and solvent extracted.

316. PIGMENTS IN COTTONSEED. Boatner, C. H. Chapter VI, pp. 213-363, Cottonseed and Cottonseed Products, Their Chemistry and Technology. Ed. by Alton E. Bailey, Interscience Publ., N. Y. 1948.

Significant contributions to the chemistry of cottonseed pigments, their occurrence and behavior in the seed, and their interconversions during processing of the seed are reviewed. A discussion of the chemical and physical properties of gossypol, its derivatives, esters, and ethers is followed by an outline of present knowledge about the more recently discovered gossypurpurin, gossycaerulin, and gossyfulvin pigments. Some data are given on pigments such as the flavones and the anthocyanins, carotenoids, chlorophyll, resin pigments, and others of an unidentified nature that have been detected in small amounts. Behavior of the pigments and pigment glands under various methods of processing cottonseed is explained with particular attention to solvent extraction and the recently developed laboratory process of gland flotation. Some possible uses for the cottonseed pigments are indicated.

291. PRE-PILOT-PLANT MIXED-SOLVENT FLOTATION PROCESS FOR SEPARATING PIGMENT GLAND FROM COTTONSEED MEATS. Vix, H. L. E.; Spadaro, J. J.; Westbrook, R. D.; Crovetto, A. J.; Pollard, E. F.; and Gastrock, E. A. J. Am. Oil Chemists' Soc. 24: 228-36. 1947.

This investigation was undertaken to develop on a pre-pilot-plant scale a special laboratory method previously found for separating flaked cottonseed into hulls, meal, and pigment glands. Data obtained during the several experimental stages through which the investigation has so far progressed are reported. Engineering and technical information is presented relative to fractionation of undefatted flakes as compared to defatted flakes, means of sufficiently disintegrating the flakes, the selection of suitable solvents, the effects of moisture in both solvents and flakes, the purification of pigment glands and meal, and the yields of these purified products. A concurrent development of the investigation was the production of high-purity pigment glands and of essentially gland-free meal in quantities sufficient to permit utilization research.

281. PIGMENT GLANDS OF COTTONSEED. III. DISTRIBUTION AND SOME PROPERTIES OF COTTONSEED PIGMENTS. Boatner, C. H.; Hall, C. M.; O'Connor, R. T.; and Castillon, L. E. Botan. Gaz. 109: 108-20. 1947.

This investigation was conducted with the twofold objective of (1) determining the extent to which the pigments of cottonseed occur inside and outside glands in the seeds and (2) acquiring information concerning the extraction of the pigments during extraction of the oil by means of different solvents. Experimental data are presented to show that all the gossypol and

gossypurpurin are segregated in the glands and constitute the only detectable pigments in the glands of the four varieties studied. Gossypol content, in contrast to that of gossypurpurin, is relatively constant, and seems to be determined by the amount of glands in the kernel. A yellow pigment detected in the oil of the extraglandular tissue was obtained free of the intraglandular pigments. It appears relatively stable and differs from other known pigments of cottonseed. Methods for the extraction and estimation of the pigments of cottonseed have been developed on the basis of the information obtained concerning their distribution and properties.

250. PIGMENT GLANDS OF COTTONSEED. II. NATURE AND PROPERTIES OF GLAND WALLS. Boatner, C. H.; Hall, C. M.; Rollins, M. L.; and Castillon, L. C. Botan. Gaz. 108: 484-93. 1947.

The structure and chemical composition of the gland wall and the behavior of the glands towards various liquids were investigated. The rigid, thick gland wall, inclosing a gelatinous suspension of the pigments, consists of from five to eight irregularly shaped, curved plates composed of cellulose apparently impregnated with pectin, hemicellulose, and other unidentified uronic acid derivatives. A layer of cutin was noted around the glands. The glands proved resistant to the action of most reagents except water and polar organic liquids of low molecular weight. The existence of a highly resistant gland wall is proposed to explain the anomaly of the relative stability of reactive polyphenolic pigments in cottonseed. The sensitivity of the glands to moisture is proposed to explain various changes during maturation of cottonseed, as well as changes during storage of moist cottonseed. Photomicrographs of cottonseed pigment glands illustrate the material.

247. THE PIGMENTS OF COTTONSEED. III. GOSSYFULVIN, A NATIVE COTTONSEED PIGMENT RELATED TO GOSSYPOL. Boatner, C. H.; O'Connor, R. T.; Curet, M. C.; and Samuels, C. S. J. Am. Chemical Soc. 69: 1268-73. 1947.

Further insight into the structure of gossyfulvin and its relationship to gossypol has been obtained. The procedure used for isolating gossyfulvin from cottonseed is outlined in a diagram. On the basis of its elementary composition, and its yields of gossypol upon acid hydrolysis, the molecular formula, $C_{34}H_{34}N_2O_8$, has been proposed for gossyfulvin. The exact correspondence of the absorption spectra of gossyfulvin and dianilinogossypol and their dissimilarity with that of gossypol have been assumed as evidence that gossyfulvin and dianilinogossypol possess identical basic structures and that both are different from the preponderant tautomeric form of gossypol. The isomerism of the three oximes formed from gossypol, dianilinogossypol, and gossyfulvin has been presented as evidence for the lability of the carbonyl groups of gossypol. Evidence has been presented indicating that the acetyl derivative formed in preponderant amounts upon acetylation of gossypol is tetra-acetoxygossypol, and that formed from gossyfulvin is a hexa-acetyl derivative.

246. THE PIGMENTS OF COTTONSEED. II. GOSSYCAERULIN, A BLUE QUINONOID GOSSYPOL DERIVATIVE OCCURRING IN COOKED COTTONSEED. Boatner, C. H.; Samuels, C. S.; Hall, C. M.; and Curet, M. C. J. Am. Chemical Soc. 69: 668-72. 1947.

A blue pigment, gossycaerulin, has been prepared by heating acidified alcoholic solutions of gossypol, and reaction conditions that favor the formation of this pigment have been determined. Purified gossycaerulin is a polar, alpha-hydroxy, quinonoid, oxidation product of gossypol. It exhibits a maximum blue color having an absorption maximum at 605 $m\mu$ in acid solution and is reversibly convertible into a nonpolar, yellow, neutral form. A yellow pigment has been detected as a byproduct during the conversion of gossypol to gossycaerulin. A purple compound resulting from the reaction of a gossycaerulin with sulfuric acid exhibits the reactions of a quinonoid compound. Gossycaerulin has been detected in cooked cottonseed, and the extent of its formation is correlated with the disappearance of gossypol.

194. THE PIGMENT GLANDS OF COTTONSEED. I. BEHAVIOR OF THE GLANDS TOWARD ORGANIC SOLVENTS. Boatner, C. H.; and Hall, C. M. Oil & Soap 23: 123-28. 1946.

The efficacy of a given solvent for the extraction of the gland pigments of cottonseed is determined not only by its solvent power for the pigments but also by its ability to attach the pigment glands and the extent to which these glands are exposed to its action, which in turn depends upon the degree of subdivision of the seed being extracted. The properties of the pigment glands have been utilized for the development of a method for mechanically fractionating cottonseed into pigment glands, embryo tissue (meal), and hull tissue. The process consists in the treatment of finely divided cottonseed with mixtures of inert liquids having densities intermediate between those of the seed parts being separated. The fractionation method has been shown to be applicable to the preparation of pigment glands and of pigment-free oil and meal from flaked cottonseed. The method is also applicable to the separation of pigment glands from defatted cottonseed.

100. GAMMA-TOCOPHEROL AS A PRECURSOR OF RED QUINOID SUBSTANCE DEVELOPED IN COTTONSEED OIL DURING OXIDATION. Swift, C. E.; Mann, G. E.; and Fisher, G. S. Oil & Soap 21(11): 317-20. 1944.

On the basis of spectrophotometric data on the characteristic spectral absorptions observed in oxidizing substrates with and without added gamma-tocopherol, it has been shown that the red color that develops on accelerated oxidation of fat substrates containing this tocopherol results from its conversion to chroman-5, 6-quinone. The chroman-5, 6-quinone thus produced possesses relatively little antioxidant activity.

86. QUANTITATIVE DETERMINATION OF EXTRACTABLE GOSSYPOL IN COTTONSEED AND COTTONSEED MEAL--A SPECTROPHOTOMETRIC METHOD. Boatner, C. H.; Caravella, Maizie; and Kyame, Lillian. Ind. Eng. Chem., Anal. Ed. 16: 566-72. 1944.

The reaction of gossypol with antimony trichloride in ether and chloroform extracts of cottonseed is specific and the reaction product is sufficiently stable to permit accurate spectrophotometric determination of the gossypol concentration of such extracts. A rapid direct method for determination of free gossypol in cottonseed is reported. Gossypol is extracted by equilibrating ground cottonseed and chloroform, treating the extract with concentrated hydrochloric acid, and applying the antimony trichloride reaction directly to the treated extract.

67. AN ORANGE-COLORED PIGMENT OF COTTONSEED. Boatner, C. H.; Caravella, Maizie; and Samuels, C. S. J. Am. Chemical Soc. 66: 838. 1944. (Republished: Oil Mill Gaz. 50(9): 15-16. 1946.)

Isolation of an orange-colored pigment from cottonseed is reported, and its properties are described. It is suggested that the occurrence of this pigment in high concentration in certain cottonseed meals may account, at least in part, for the frequently observed lack of correlation of gossypol content with toxicity of these meals when the gossypol is determined as the dianilino compound.

64. THE PIGMENTS OF COTTONSEED. Boatner, C. H. Oil & Soap 21(1): 10-15. 1944.

A method for the purification of gossypol has been developed that yields material differing in optical properties from those previously reported for gossypol preparations. A colorimetric test for extractable gossypol has been described. It has been shown that cottonseed contains at least three pigments in addition to gossypol. Some of the properties of the three pigments have been reported. One of these newly detected pigments, gossypurpurin, has been shown to be the substance that, mixed with gossypol, constitutes the so-called "red gossypol" of Podol'skaja. The frequently reported water-dispersible blue pigment of cottonseed has been

shown to be either a complex of gossypol, gossypurpurin and protein, or a mixture of two protein-pigment complexes. Its dissociation into the two pigments and protein has been accomplished.

GOSSYPOL AND OTHER PIGMENTS - PATENTS

2460. PROCESS FOR RECOVERY OF GOSSYPOL FROM COTTONSEED GUMS. Pons, W. A., Jr.; Pominski, Joseph; and King, W. H. U. S. Pat. No. 3,062,876, November 6, 1962.

Processes for isolating gossypol and, particularly, a process for recovering gossypol from cottonseed gum are described. Cottonseed gum is a low-cost byproduct obtained by water-washing crude cottonseed oil.

749. WATER-SOLUBLE COMBINATION PRODUCTS OF GOSSYPOL AND AMINO ACIDS. Altschul, A. M.; and Castillon, L. E. U. S. Pat. No. 2,582,949, January 22, 1952.

The combination products are toxic to goldfish but not to rats or mice, and are produced by dissolving gossypol and an amino acid, such as lysine, in a basic aqueous medium, (neutralizing the solution and freezing the medium) and removing the water by sublimation.

748. WATER-SOLUBLE COMBINATION PRODUCTS OF GOSSYPOL AND PROTEINS. Altschul, A. M.; and Castillon, L. E. U. S. Pat. No. 2,581,342, January 8, 1952.

The products are toxic to goldfish. These materials are prepared by dissolving gossypol and proteins, such as peanut or casein proteins, in a basic aqueous medium, (neutralizing the solution and freezing the medium) and removing the water by sublimation.

679. WATER-DISPERSIBLE COMBINATION PRODUCTS OF GOSSYPOL AND CARBOHYDRATES. Altschul, A. M.; and Castillon, L. E. U. S. Pat. No. 2,563,808, August 14, 1951.

Mixing gossypol and carbohydrates (potato starch, dextrose) in basic aqueous solution, neutralizing, and drying without heat produces water-dispersible combination products having physiological properties different from gossypol.

428. HEAVY GRAVITY LIQUID SEPARATION OF COTTONSEED. Boatner, C. H.; Hall, C. M.; and Merrifield, A. L. U. S. Pat. No. 2,482,141, September 20, 1949.

This patent describes a process for separating the pigments from a cottonseed product containing substantially intact pigment glands embedded in the seed embryo tissues. The process comprises treating the product to disintegrate the embryo tissue and release the intact pigment glands, mixing with a liquid that does not substantially rupture the pigment glands and that has a density greater than that of the pigment glands to be separated and less than that of the remaining parts, and then settling the mixture so the pigment glands float to the top where they can be removed.

SPECIAL INVESTIGATIONS - CYCLOPROPENOIDS

3262. METHODS FOR THE DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. VII. THE DILUTION-HBr-TITRATION TECHNIQUE AS A GENERAL METHOD. Magne, F. C.; Harris, J. A.; Pittman, R. A.; and Skau, E. L. J. Am. Oil Chemists' Soc. 43: 519-24. 1966.

The stepwise HBr titration method for the cyclopropenoid analysis of cottonseed oils is subject to serious inaccuracies when applied to samples containing higher cyclopropenoid concentrations, particularly if they contain appreciable amounts of alumina-adsorbable materials. A modification of the method is described that eliminates these sources of error. Its validity

has been established by the analysis of a wide range of synthetic compositions including compositions containing massive amounts of interfering HBr-reactive substances and other alumina-adsorbable materials. The method with further modification can be used to analyze glycerides with the same high degree of accuracy.

3197. SOME PHYSIOLOGICAL PROPERTIES OF HALPHEN-POSITIVE COTTONSEED OILS. Frampton, V. L.; Kuck, J. C.; Pepperman, A. B., Jr.; Pons, W. A., Jr.; Watts, A. B.; and Johnston, C. Poultry Sci. 45: 527-35. 1966.

Investigations of the properties of fresh and stored shell eggs produced by hens on rations containing cottonseed oils have established that the in vivo reaction in the stearic acid ←· · · · → oleic + linoleic acids then is driven abnormally to the left. The degree of disturbance in the stearic, oleic, and linoleic acids in yolk fat was observed to be related to the concentration of malvalic acid in the ingested cottonseed oils. Halphen-negative cottonseed oils, produced by bleaching cottonseed oil with sulfurous acid-treated alumina, did not have this property of disturbing the distribution of these three fatty acids in egg yolk fat. Coefficients of variation for fatty acids in cottonseed oils were determined.

3122. A SIMPLIFIED PROCESS FOR THE ELIMINATION OF THE HALPHEN-TEST RESPONSE IN COTTONSEED OILS. Rayner, E. T.; Brown, L. E.; and Dupuy, H. P. J. Am. Oil Chemists' Soc. 43: 113-15. 1966.

Since certain biological effects have been ascribed to the cyclopropenoids that give a positive response to the Halphen test, processes were explored to eliminate this response from cottonseed oils. Heating alkali-refined, Halphen-positive cottonseed oils in a modified laboratory deodorizer in the presence of cottonseed oil fatty acids, capric acid, citric acid, or phosphoric acid was found to be an effective method of eliminating this response. These treated Halphen-negative cottonseed oils, the untreated Halphen-positive cottonseed oil, and a corn oil control were incorporated into rations fed to different groups of laying hens. Hens that ingested either the Halphen-negative cottonseed oil or the corn control produced normal eggs, whereas hens fed the Halphen-positive cottonseed oil produced eggs with pink whites, decreased pH of whites, increased pH of yolks on storage, and lower ratios of oleic acid to stearic acid in the yolk lipids. The simple processes presented--particularly the use of cottonseed oil fatty acids--appear to offer a practical means of inactivating the cyclopropenoids in cottonseed oil and thus eliminating the biological effects attributed to them.

3121. CYCLOPROPENOID FATTY ACID CONTENT AND FATTY ACID COMPOSITION OF CRUDE OILS FROM TWENTY-FIVE VARIETIES OF COTTONSEED. Bailey, A. V.; Harris, J. A.; Skau, E. L.; and Kerr, T. J. Am. Oil Chemists' Soc. 43: 107-110. 1966.

The cyclopropenoid acid content of oils extracted from 22 commercial varieties and 3 botanical species of cottonseed has been determined. The malvalic acid content determined by HBr titration varied from a low of 0.56 percent to a high of 1.17 percent. Iodine values of the oils ranged from 96.8 to 111.6. No definite correlation could be established between iodine value and malvalic acid content. Equations for regression lines for the major acids have been calculated from plots of fatty acid composition versus iodine value. The high degree or correlation suggests that for commercial oils the fatty acid composition can be estimated from the iodine value. Oils of the 3 experimental types of different species showed wide variations in fatty acid composition and represented many of the maximum and minimum values reported.

3023. PREPARATION OF METHYL MALVALATE FROM STERCULIA FOETIDA SEED OIL. [PL-480 GRANT]. Fogerty, A. C.; Johnson, A. R.; Pearson, J. A.; and Shenstone, F. S. (C. S. I. R. O. Division of Food Preservation, Ryde, N. S. W., Australia) J. Am. Oil Chemists' Soc. 42, 885-87. 1965.

Methyl malvalate has been prepared from mixed methyl esters of Sterculia foetida seed oil fatty acids by reversed-phase liquid-liquid partition chromatography and confirmatory evidence adduced for its structure.

2970. METHODS FOR THE DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. VI. A DIRECT INFRARED ABSORPTION METHOD. Bailey, A. V.; Boudreaux, G. J.; and Skau, E. L. J. Am. Oil Chemists' Soc. 42 : 637-38. 1965.

A simple rapid method for estimation of the cyclopropenoid content of glycerides and methyl esters is described, based upon the measurement of the characteristic infrared absorptivity of cyclopropenoids at 9.9μ. Autoxidation products do not interfere, and the sample can be recovered. Equations are given for the calculation of the cyclopropenoid content of both glycerides and methyl esters.

2917. METHODS FOR THE DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. V. A SPECTROPHOTOMETRIC METHOD FOR COTTONSEED OILS BASED UPON THE HALPHEN-TEST REACTION. Bailey, A. V.; Pittman, R. A.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 42 : 422-24. 1965.

A spectrophotometric method of analysis for the quantitative estimation of cyclopropenoid fatty acids in cottonseed oil, based upon the Halphen test reaction, is described. Various parameters involved in the reaction were investigated and two pigment fractions responsible for the characteristic Halphen test cherry-red color isolated. The method is applicable to relatively small amounts of sample material. The average deviation from the actual cyclopropenoid acid contents as determined by the stepwise HBr titration method was less than $\pm$ 0.02 percent in both the refined and crude oil series.

2903. ANALYSIS OF CYCLOPROPENOID AND CYCLOPROPANOID ACIDS IN FATS AND OILS. Magne, F. C. J. Am. Oil Chemists' Soc. 42 : 322-36. 1965.

The analysis of cyclopropenoid acids may be considered, from a historical standpoint, to have started with the discovery of the Halphen test. Although this test as originally conceived was utilized as a means of detecting adulteration of premium edible oils with cottonseed oil, it has since been shown to be a characteristic test for cyclopropenoid fatty acids and has been adapted with various modifications as a quantitative colorimetric test for these substances. More recently, spectrophotometric methods particularly in the infrared region have been applied to the analysis of these substances. The 9.8μ band characteristic of the cyclopropane, and the 9.91μ band characteristic of the cyclopropene group, as well as the 11.0μ band characteristic of some of the noncyclic degradation derivatives have been used. Gas-liquid chromatography (GLC) has been applied to the methyl esters of cyclopropanoid and hydrogenated cyclopropenoid acids. The reactivity of the cyclopropene ring toward hydrohalogens has been the basis of several analytical methods developed for use with cyclopropene acid-containing oils. Both aqueous and nonaqueous solutions of hydrohalogens have been employed. The hydrohalogenation methods are the most precise methods currently available for these analyses, but only GLC has the inherent potential of identifying the specific cyclopropenoid or cyclopropenoids involved.

2887. CYCLOPROPENOID FATTY ACID CONTENT AND FATTY ACID COMPOSITION OF CRUDE COTTONSEED OILS FROM SUCCESSIVE SOLVENT EXTRACTIONS. Bailey, A. V.; Pons, W. A., Jr.; and Skau, E. L. J. Am. Oil Chemists' Soc. 42 : 173-76. 1965.

The fatty acid composition and properties of six fractions of oil successively extracted from cottonseed meats have been investigated. The cyclopropenoid fatty acid concentration increased regularly from 0.30 to 1.06 percent a 3.5 -fold increase. This suggests that the cyclopropenoid constituents of the oil in the seed are less accessible to the solvent. The linoleic acid concentration decreased from 56.3 to 53.1 percent accounting for a slight reduction in iodine value (I. V.). The first two fractions had a markedly lower phosphatide content than the remaining fractions.

2791. REVIEW OF THE CHEMISTRY OF CYCLOPROPENE COMPOUNDS. Carter, F. L.; and Frampton, V. L. Chem. Rev. 64: 497-525. 1964.

The recent discovery of naturally occurring fatty acids that contain the cyclopropene ring has intensified the interest in cyclopropene compounds. The syntheses, chemical reactions, and physical properties of cyclopropene and synthetic cyclopropene compounds are described and compared in this review.. A discussion of the controversial Feist's acid is included as it was considered to be a cyclopropene compound for more than a half century. The physical properties of the cyclopropene derivatives obtained from the literature through 1963 are listed in a table for both comparative purposes and for general information. Considerable attention is given to the naturally occurring cyclopropene fatty acids, sterculic and malvalic acids, their isolation, proofs of their structures, chemical reactions, and their physiological properties. The Halphen test is described in detail, as a positive response is given by vegetable oils containing cyclopropene fatty acids. Other methods of analysis are described. Closely related, naturally occurring cyclopropane fatty acids are briefly mentioned.

2780. CYCLOPROPENYL COMPOUNDS AS SULFHYDRYL REAGENTS. Ory, R. L.; and Altschul, A. M. Biochem. Biophys. Res. Commun. 17(1): 12-16. 1964.

In vitro inhibition of the lipase of Ricinus communis, a sulfhydryl-sensitive enzyme, by Sterculia foetida oil and sterculene has been demonstrated. The inhibition can be reversed by excess cysteine if added before incubation of enzyme and inhibitor, but not if added later. This reaction between CPA and a sulfhydryl enzyme suggests that CPA may act physiologically as SH-enzyme inhibitors; especially enzymes involved in lipid metabolism.

2710. METHODS FOR THE DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. IV. APPLICATION OF THE STEP-WISE HBr TITRATION METHOD TO THE ANALYSIS OF REFINED AND CRUDE COTTONSEED OILS. Harris, J. A.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 41: 309-11. 1964.

A method is described for the determination of cyclopropenoid fatty acids in refined and crude cottonseed oils to within 0.01 percent. It is based upon a step-wise hydrogen bromide (HBr) titration at 3° C and 55° C after removal of interfering substances by adsorption on activated alumina. Highly oxidized cottonseed oils first must be converted to methyl esters.

2683. METHODS FOR DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. INFRARED ABSORPTION METHOD. Magne, F. C.; Bailey, A. V.; McCall, E. R.; Miles, S. H.; and Skau, E. L. Anal. Chem. 36: 681-82. 1964.

An infrared method is described for the estimation of long-chain cyclopropenoid fatty acid derivatives. It is based upon the measurement of the absorptivity, α, of the sample at 11.05μ after shaking with concentrated hydrochloric acid. The percentage of cyclopropenoid moiety, x, calculated as sterculic acid, can be determined from the equation $x = 518.56\alpha - 5.186$.

2645. METHODS FOR THE DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. II. A STEPWISE HYDROGEN BROMIDE TITRATION METHOD FOR CYCLOPROPENOID AND EPOXY DERIVATIVES. Harris, J. A.; Magne, F. C.; and Skau, E. L. J. Am. Oil Chemists' Soc. 40: 718-20. 1963.

A rapid titration method id described for the quantitative determination of both cyclopropenoid and epoxy fatty acid derivatives in mixtures. It was found that epoxy compounds can be titrated selectively with Durbetaki reagent at 3° C without interference from cyclopropenoid derivatives. Cyclopropenoid derivatives can be titrated much more rapidly to a much sharper end point at 55° C than at room temperature. Thus, mixtures can be analyzed by first titrating at 3° C to determine the epoxy compounds and then continuing the titration at 55° C to determine the cyclopropenoid components.

2644. METHODS FOR THE DETERMINATION OF CYCLOPROPENOID FATTY ACIDS. I. AQUEOUS HYDROCHLORIC ACID METHOD. Magne, F. C.; Harris, J. A.; and Skau, E. L. J. Am. Oil Chemists' Soc. 40: 716-17. 1963.

An analytical method is described for the estimation of long-chain cyclopropenoid fatty acid derivatives. It is based upon the quantitative addition of a molecule of hydrogen chloride at the cyclopropene ring when the sample is shaken with concentrated hydrochloric acid. The cyclopropenoid content can be calculated, as sterculic acid, from the increase in chlorine content. Epoxy compounds and hydroperoxides interfere and must be removed by one of the accepted pretreatment methods.

CYCLOPROPENOIDS - PATENTS

2751. PROCESS FOR REMOVING THE HALPHEN TEST RESPONSE FROM COTTONSEED OIL. Rayner, E. T.; Heinzelman, D. C.; and Dupuy, H. P. U. S. Pat. No. 3,135,775. June 2, 1964.

This invention relates to a process for rmoving the Halphen test response from cottonseed oil and has as its object the production of cottonseed oils that react negatively to the Halphen test.

SPECIAL INVESTIGATIONS - AFLATOXIN

3313. INACTIVATION AND REMOVAL OF AFLATOXIN. Dollear, F. G.; and Gardner, H. K., Jr. Proc. Fourth Natl. Peanut Res. Conf. (Tifton, Ga., July 14-15, 1966), 72-81. 1966.

Investigations are directed toward the development of practical means for the deactivation, destruction, and/or removal of aflatoxin from oilseed products, particularly peanut and cottonseed meals. Some approaches include: (a) physical means of separation of sound from contaminated seeds; (b) inactivation or destruction of alflatoxin by heat and moisture, or by chemical treatment; and (c) removal by solvent extraction in conventional equipment. A number of chemicals have been screened, and these are listed according to the results obtained. Several appear promising.

3256. PRODUCTION OF AFLATOXINS B_1 AND G_1 BY ASPERGILLUS FLAVUS IN A SEMI-SYNTHETIC MEDIUM. Davis, N. D.; Diener, U. L.; and Eldridge, D. W. Appl. Microbiol. 14: 378-80. 1966.

Isolates of Aspergillus flavus produced 0.2 to 63 mg. of aflatoxins B_1 and G_1 per 100 ml. in a nutrient solution consisting of 20 percent sucrose and 2 percent yeast extract. Various factors influencing the fermentation were studied. The maximal amount of toxin was produced by American Type Culture Collection culture 15548 in 1-liter flasks containing 100 ml. of medium incubated as stationary cultures for 6 days at 25° C.

3203. DETERMINATION OF AFLATOXINS IN AGRICULTURAL PRODUCTS: USE OF AQUEOUS ACETONE FOR EXTRACTION. Pons, W. A., Jr.; Cucullu, A. F.; Lee, L. S.; Robertson, J. A.; Franz, A. O.; and Goldblatt, L. A. J. Assoc. Offic. Agr. Chemists 49: 554-62. 1966.

An analytical procedure originally developed for the determination of aflatoxins in cottonseed products has been modified for application to many agricultural materials. Aflatoxins are rapidly extracted free of lipid contamination with 70 percent acetone. Many interfering pigments are removed from the crude extract by precipitation as insoluble lead derivatives. Aflatoxins are transferred into chloroform, and the chloroform extract is further purified with silica gel. The procedure was compared with four recently proposed methods for the analysis of peanut products. This technique is capable of detecting as little as 0.3 p. p. b. of aflatoxin B_1 in peanuts and peanut butters. Adequate recovery of added aflatoxins was obtained when this method was applied to numerous different agricultural products.

3151. ELABORATION OF AFLATOXIN ON COTTONSEED PRODUCTS BY ASPERGILLUS FLAVUS. Mayne, R. Y.; Pons, W. A., Jr.; Franz, A. O., Jr.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 43 : 251-53. 1966.

In controlled laboratory experiments heat sterilized and unautoclaved glanded and glandless whole cottonseed or decorticated kernels and sterilized cottonseed meals were found to be utilized as substrates by an aflatoxin-elaborating strain of A. flavus with the production of high levels of aflatoxins B_1, G_1, and G_2. Gossypol pigments in cottonseed products are apparently not a barrier to either mold invasion or aflatoxin production. Cottonseed hulls, lint cotton, and cottonseed linters were found to be poorly utilized as substrates for either mold growth or aflatoxin production.

3119. DETERMINATION OF AFLATOXINS IN INDIVIDUAL PEANUTS AND PEANUT SECTIONS. Cucullu, A. F.; Lee, L. S.; Mayne, R. Y.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 43 : 89-92. 1966.

Subsamples of a given lot of peanuts may vary greatly in aflatoxin content due to extreme variability in the degree of contamination of individual kernels. A micro method, adapted from the aqueous acetone procedure recently proposed by Pons and Goldblatt for the determination of aflatoxins in cottonseed products, was developed to permit accurate determination of aflatoxins in individual kernels and kernel sections. Use of this procedure permitted the topographic distribution of aflatoxins within single kernels to be mapped and indicated that the toxins are not uniformly distributed within contaminated kernels, even when the kernel contains a high level of aflatoxins. Although wrinkling or discoloration sometimes indicated that a kernel was contaminated, this type of physical damage was not found to be a reliable indication of aflatoxin content. Also, it was noted that a few apparently sound and mature kernels contained high levels of aflatoxins.

3115. AFLATOXIN. Goldblatt, L. A. Assoc. Food Drug Officials U. S. Quart. Bull. 29 : 158-69. 1965.

Molds in feeds have been a problem for a great many years. The developments in 1960 relative to the "Turkey X" disease in poultry dramatized and focused attention on the problem of mold toxins, especially aflatoxin, and gave impetus to mold toxin research. The Southern Regional Research Laboratory is conducting one part of a comprehensive Department program of research on mycotoxins that embraces many agricultural commodities. Its broad objectives are: (1) to develop analytical methodology; (2) to determine the effect of graded levels of aflatoxin on various experimental animals, including farm animals; (3) to find practical methods for preventing or at least minimizing mold growth in our commodities; and (4) to develop processes for removing or destroying toxins in those lots of our commodities that may have been contaminated with toxin-producing molds. Some of the results obtained from this work will be presented. Excellent liaison has been established and is being maintained by the Department with affected industry groups, such as the research committees of the National Peanut Council, the National Cottonseed Products Association and with the Food and Drug Administration. The presence of mold toxins is potentially the most serious quality problem that faces the producers, manufacturers, and handlers of food and feed products. It could become a problem for all agricultural commodities, not just cottonseed and peanuts. The problem is an important one, but it is getting much research attention.

2998. THE MYCOTOXIN PROBLEM. Goldblatt, L. A. Oil Mill Gaz. 70 (3) : 16-22. 1965.

Molds in feeds have been a problem for a great many years. The developments in 1960 relative to the "Turkey X" disease in poultry dramatized and focused attention on the problem of mold toxins and gave impetus to mold toxin research. The Southern Regional Research Laboratory is conducting one part of a comprehensive U. S. Department of Agriculture program of research on mycotoxins that embraces many agricultural commodities. Its broad objectives are: (1) to develop analytical methodology; (2) to determine the effect of graded levels of aflatoxin on

various experimental animals, including farm animals; (3) to find practical methods for preventing or at least minimizing mold growth in our commodities; and (4) to develop processes for removing or destroying toxins in those lots of our commodities that may have been contaminated with toxin-producing molds. Some of the results obtained are presented. Excellent liaison is maintained by the Department with affected industry groups, such as the research committees of the National Peanut Council, The National Cottonseed Products Association, and with the Food and Drug Administration. The presence of mold toxins is potentially the most serious quality problem that faces the producers, manufacturers, and handlers of food and feed products. It could become a problem for all agricultural commodities, not just cottonseed and peanuts. The problem is an important one, but it is getting much research attention.

2968. REMOVAL OF AFLATOXIN FROM PEANUT PRODUCTS WITH ACETONE-HEXANE-WATER SOLVENT. Goldblatt, L. A. Intern. Symp. "Mycotoxins in Foodstuffs," Cambridge, Mass., 1964 : 261-63. (Publ. 1965.)

A solvent mixture composed of acetone, hexane, and water is potentially applicable to solution of the aflatoxin problem. Four different areas of possible usefulness of this solvent mixture are described. These include: (1) extraction solvent for analysis, (2) removal of aflatoxin from peanut meal and other oilseed meals, (3) conversion of peanuts to aflatoxin-free oil and (4) removal of aflatoxin from whole peanuts. The value of this solvent mixture for analysis has been established and its potential utility in commercial processing is indicated.

2931. THE DETERMINATION OF AFLATOXINS IN COTTONSEED PRODUCTS. Pons, W. A., Jr.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 42 : 471-75. 1965.

A rapid and simplified procedure is proposed for the determination of aflatoxins B_1, B_2, G_1, and G_2 in cottonseed products. The method involves extraction of aflatoxins essentially free of lipid contamination by equilibrium extraction with 70 percent acetone. Interfering gossypol pigments are removed from the aqueous acetone extract by precipitation as insoluble lead salts. Aflatoxins in the centrifugate are quantitatively separated from excess lead salts, residual pigments, and carbohydrates by extraction into chloroform to yield final extracts that are low in total solids and pigmentation for thin-layer chromatographic (TLC) analysis on silica gel. The procedure is sensitive to about 1 p. p. b. in cottonseed meats and 4 p. p. b. in cottonseed meal.

2930. ASSAY OF AFLATOXIN IN PEANUTS AND PEANUT PRODUCTS USING ACETONE-HEXANE-WATER FOR EXTRACTION. Robertson, J. A., Jr.; Lee, L. S.; Cucullu, A. F.; and Goldblatt, L. A. J. Am. Oil Chemists' Soc. 42 : 467-71. 1965.

A quantitative method is described for the assay of aflatoxin in peanut products. The procedure involves extraction of aflatoxin from the sample with a homogeneous acetone-hexane-water solvent mixture followed by purification of the extract by phasic extraction of the aflatoxin with aqueous sodium chloride and then with chloroform. The purified chloroform extract is analyzed by thin-layer chromatography by comparison of the intensity of fluorescence of any aflatoxin with the intensity of a known standard. The aflatoxin analyses of peanuts were found to be very variable because of sampling methods. This variability has been greatly reduced by finely grinding and thoroughly mixing 2 kg. of the sample before removal of an aliquot for assay. The method is sensitive to approximately 2 parts per billion.

2919. GIBBERELLIC ACID-INDUCED LIPASE AND α-AMYLASE FORMATION AND THEIR INHIBITION BY AFLATOXIN. Black, H. S.; and Altschul, A. M. Biochem. Biophys. Res. Commun. 19 : 661-64. 1965.

The effects of gibberellic acid (GA) and aflatoxin on enzyme activities, notably lipase and α-amylase, in excised tissues of seeds were studied. Aflatoxin was shown to inhibit GA-induced

formation in cottonseed as well as GA-induced -amylase formation in the aleurone layer of barley. It was suggested that aflatoxin is an inhibitor of protein synthesis.

2910. BIBLIOGRAPHY ON AFLATOXIN FROM 1960. Skau, D. B. U. S. Dept. Agr.; Agr. Res. Service, Sou. Util. Res. and Dev. Div., 40 pp. 1965.

A list of references relating to the occurrence, production, properties, and effects of aflatoxin.

2840. REVIEW OF RESEARCH ON AFLATOXIN AT THE SOUTHERN REGIONAL RESEARCH LABORATORY. Goldblatt, L. A. Proc. Natl. Peanut Res. Conf., 3rd: 128-33. 1964.

Current and planned research on aflatoxin at the Southern Regional Research Laboratory as related to peanuts and cottonseed is discussed. Reference is made to extraction of cottonseed meals in connection with a study of the trout hepatoma problem, preparation of peanut products for use in long-term rat feeding trials, development of analytical methodology, potential utility of acetone-hexane-water solvent mixtures to prepare aflatoxin-free products, surveys of prevalence of aflatoxin in domestic stocks of peanuts, and investigation of other fungal flora of cottonseed and peanuts to determine if they produce toxins harmful to animals. Reference is made to cooperation with the Pharmacology Laboratory at the Western Utilization Research and Development Division and to research on aflatoxin by contract and with PL-480 funds.

2702. BIBLIOGRAPHY ON AFLATOXIN FROM 1960. Skau, D. B.; Robertson, J. A., Jr.; and Mayne, R. Y. U. S. Dept. Agr., Agr. Res. Service, Sou. Util. Res. and Dev. Div., 15 pp. 1964.

A list of references relating to the occurrence, production, properties, and effects of aflatoxin. (Superseded by No. 2910 and no longer available.)

1379. LIPOLYTIC MICROORGANISMS ASSOCIATED WITH PRIME AND DETERIORATED COTTONSEED. Mayne, R. Y. Appl. Microbiol. 4: 263-69. 1956.

The general quantity of microflora, the numbers of lipolytic microorganisms, and the identities of most of the lipolytic microorganisms contained in samples of cottonseed with different amounts of free fatty acids in the oil of the seed have been determined. Most (nine-tenths) of the numbers of cottonseed samples with large contents of free fatty acids in the oil contained large numbers, at least 10,000 per gram, of lipolytic molds or lipolytic bacteria. None of the samples with low amounts of free fatty acids in the oil contained large numbers of lipolytic molds, but some contained large numbers of lipolytic bacteria. Some of the lipolytic bacteria in these good quality seed were included in a group of lipolytic organisms that were either weak or variable in their activity. The lipolytic molds were identified as Penicillium lanosum, P. citrinum, P. roseopurpureum, P. pusillum; Aspergillus flavus, A. awamori, A. luchuensis, A. terreus two varieties; Fusarium moniliforme, F. nivale, F. roseum; F. oxysporum, F. episphaeria; a Diplodia sp., a Hormodendrum sp.; a Ceratostomella sp.; and some unidentified species. The lipolytic bacteria were Xanthomonas-like organisms, Pseudomonas aeruginosa; P. ovalis, P. reptilivorous, other Pseudomonas species Bacillus subtilis, B. pumilus, and a few unidentified gram-positive bacilli.

1378. LIPOLYTIC ACTIVITY OF VARIOUS MICROORGANISMS ISOLATED FROM COTTONSEED. Mayne, R. Y. Appl. Microbiol. 4: 270-73. 1956.

Quantitative tests of the activities of pure cultures of lipolytic microorganisms isolated from samples of cottonseed revealed large differences in the quantities of fat utilized and in the accumulation of free fatty acids. For convenience, the lipolytic organisms were arranged in five groups as follows: (1) The Xanthomonas-like bacteria that used large amounts of neutral fat and accumulated large amounts of fatty acid; (2) a group of molds, mostly of the genera Aspergillus and Penicillium, that used large amounts of neutral fat and accumulated moderate amounts of fatty acids; (3) a group of strains of Aspergillus flavus that used large amounts of neutral fat but accumulated only small amounts of fatty acids; (4) a group of species of various

molds, and Pseudomonas species that were moderately lipolytic; and (5) a group, consisting mainly of gram positive rods that was only weakly lipolytic. Organisms of very strong lipolytic ability were found generally in seed of bad quality. Also the rate of lipolysis seemed to correlate with the total amount of neutral fat used and free fatty acids produced.

LINTERS* AND BATTING

3220. DRYING AND CURING CHEMICALLY TREATED COTTON BATTING. Holzenthal, L. L.; Haydel, C. H.; and Knoepfler, N. B. U. S. Dept. Agr. ARS 72-45, 14 pages. 1966.

Part I of this paper describes preliminary determination of conditions for the drying and curing of cotton batting spray treated with a resin and latex formulation. Part II reports on the design, construction, and operation of the pilot-plant continuous oven used for drying and curing.

3148. PROGRESS IN THE COMMERCIALIZATION OF IMPROVED COTTON BATTING THROUGH DEVELOPMENTAL RESEARCH. Knoepfler, N. B.; and Koenig, P. A. Cotton Gin & Oil Mill Press 67(8) : 7-8, 10. 1966.

The situation of cotton batting in competition with polyurethane foam and other synthetic cushioning materials in the early 1960's is described to set the background for the cooperative research effort to upgrade cotton batting products. The research problem is defined, and the development of a process for the production of superior cotton batting product called Cotton Flote is traced. The rapid utilization of the research data by the batting industry, and the acceptance of Cotton Flote by the automobile industry based upon the performance of research samples are discussed.

2988. TEST METHODS FOR EVALUATING COTTON FLOTE, A NEW COTTON BATTING PRODUCT. Knoepfler, N. B. Bedding 89(1): 35-38. 1965.

Because the cotton batting industry has no widely accepted performance standards or test procedures to objectively measure the characteristics of the products, it was necessary during the course of the research leading to the development of Cotton Flote to devote some research effort to measure product performance and quality. A number of test methods for the evaluation of Cotton Flote are described and some of their shortcomings and their objectivity are discussed. Liaison with the National Cotton Batting Institute to develop appropriate test procedures and workable standards for the new product are noted.

2607. SIMPLE TEST DETERMINES COMPRESSIONAL SET OF COTTON BATTS. Gentry, W. T., Jr.; Haydel, C. H.; and Knoepfler, N. B. Bedding 85(2): 41-44, 46. 1963.

A test instrument and a procedure for the use of the instrument in measuring the compressional set taken by cotton batting samples have been developed. This instrument has been designed so that tests can be carried out at any desired temperature and relative humidity. The technique and the procedure for the test have been evaluated for duplicability. The following procedure is recommended: After die cutting, the samples should be equilibrated under standard conditions of temperature and relative humidity for at least 24 hours. Test should be carried out both at 60 percent and 100 percent relative humidity, and the initial compressional sets should be calculated. Where recovery performance characteristics are desired, the samples should be allowed to recover from the loadings at ambient conditions by placing them on the laboratory bench. Measurements should be taken at intervals up to 1 hour, and the recovery or compressional set, or both, calculated for each reading.

* Earlier work, largely directed toward the manufacture of nitrocellulose (guncotton) from linters has been omitted as having no current interest to processors of cottonseed.

2503. COTTON RESEARCH AIMS AT RECAPTURING BATTING MARKETS. Knoepfler, N. B.; Vix, H. L. E.; Gardner, H. K.; and Patton, E. L. Bedding Magazine 84 (3): 40-44, 46, 48, 50. 1963.

Research to improve the resilience, dimensional stability, and the coherence of cotton batting was undertaken by the SURDD in cooperation with the Textile Waste Association, the National Cottonseed Products Association, the National Cotton Batting Institute, and the Foundation for Cotton Research and Education. Research to date has established the feasibility of applying certain resins and latexes to cotton batting while it is in the web form by spraying the reactants onto the cotton on the apron discharge from the garnett. By proper selection of the resins and latexes, certain systems have been derived that are compatible with each other in the solution suspension to be sprayed. Under these conditions a single spray operation accomplished the application of the two components needed to treat the batting. Drying and curing of the products are required. Restraint of the batting during drying and curing permits the control of the density and improves dimensional stability, and molded products are possible. Indications are that a range of products having different resiliences and densities can be specifically designed for particular end-use applications. Results of the first 9 months of this research work are reported.

1646. CHEMICAL MODIFICATION OF COTTON LINTERS FOR PAPERMAKING. Spadaro, J. J.; Janssen, H. J.; Vix, H. L. E.; and Simons, F. L. Tappi 41: 674-79. 1958.

A study to determine the papermaking characteristics of chemically modified cotton linters was undertaken as a cooperative project by the SURDD and Crane and Company. Samples of cyanoethylated and of hydroxyethylated cotton linters at various degrees of substitution were prepared by SURDD, and the laboratory beater was evaluated by Crane and Company. The paper describes the methods used for cyanoethylation with acrylonitrile and for hydroxyethylation with ethylene oxide. Results of the beater-evaluation tests are given graphically to show the effect of the degree of treatment on the strength characteristics of the prepared handsheets and on beating time and hydration. No appreciable improvements were obtained by the cyanoethylation treatment. The hydroxyethylation treatment, however, gave some very interesting results in that folds (Schopper) of up to 14,000 were obtained, and freeness was decreased to 115 at a beating time of 120 minutes. Tests for the control sample showed 500 folds and a freeness of 220 at a beating time of 180 minutes.

CONFERENCES

COTTONSEED PROCESSING AS RELATED TO NUTRITIVE VALUE OF THE MEAL. No Proceedings are available for any of these five Conferences, which were held on the following dates:

November 13-14, 1950
November 5-7, 1951
November 9-11, 1953
January 14-16, 1957
January 19-20, 1959

COTTONSEED PROTEIN FOR ANIMAL AND MAN
November 14-16, 1960
Proceedings, ARS 72-24, January 1962

SEED PROTEIN
January 21-23, 1963

COTTONSEED PROTEIN CONCENTRATES
January 15-17, 1964
Proceedings, ARS 72-38, April 1965

These three latter conferences were international in scope and reflected the growing interest in cottonseed and other plant products as a source of protein for human nutrition as well as animal feeding. Leading authorities from various parts of the free world participated and presented the latest developments in the processing and use of seed proteins as human food and the possibilities for these products to make up the worldwide protein deficit.

COTTONSEED PROCESSING CLINICS

The first Cottonseed Processing Clinic was held in New Orleans in 1952 and has been held annually ever since. It is sponsored by the Mississippi Valley Oilseed Processors Association, Inc. (formerly Valley Oilseed Processors Association) and the SURDD. Papers and discussions at these Clinics reflect the changing problems and interests of the industry. These papers, discussions, and other data pertinent to the meeting are incorporated in Proceedings, which are available for distribution so long as the supply lasts. Proceedings of the earlier conferences in this series are no longer available.

REVIEWS OF RESEARCH

3302. COTTONSEED'S ROLE IN A HUNGRY WORLD. Lambou, M. G.; Shaw, R. L.; Decossas, K. M.; and Vix, H. L. E. Econ. Botany 20: 256-67. 1966.

Cottonseed, grown in a substantial number of developing countries, is an important source of vegetable protein for supplemental feeding of humans. Although the percentage of flour for human consumption currently being produced from this cottonseed still does not provide the total needs for supplemental protein feeding, it is believed that improved technology and handling of this important crop can go a long way toward meeting the critical protein shortage. At least two products have been made containing cottonseed flour and these are being supplied commercially for supplemental human feeding in a number of Latin American countries.

3266. CONTRIBUTIONS OF THE SOUTHERN REGIONAL RESEARCH LABORATORY TO THE ADVANCEMENT OF THE COTTONSEED OIL MILLING INDUSTRY. Gastrock, E. A. Oil Mill Gaz. 71(4): 22-29. 1966.

Research at the SURDD cottonseed and cottonseed products during the past 26 years is reviewed, showing the numerous contributions made to the advancement of the cottonseed industry. Areas covered include research on gossypol, solvent extraction, cotton batting, improved cottonseed meal for animal nutrition, and the development of new products, such as coatings and plasticizers. Current research is also outlined with regard to its importance to the present and future. A list of 113 major technical publications reporting on the work of the Southern Division is appended.

3214. RECENT DEVELOPMENTS IN OILSEED RESEARCH AT THE SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION. Kopacz, B. M.; Hoffpauir, C. L.; and Dollear, F. G. Cotton Gin & Oil Mill Press 67(15): 7-8, 25-29. 1966.

Recent oilseed research at the SURDD is reviewed. A list of 87 publications reporting on this research, with titles and literature references, is given for those wanting further details on a specific subject. Commodities covered are cottonseed, peanuts, and tung, and the areas include processing and product development, proteins, mycotoxins, the cyclopropenoids in cottonseed, and investigations of the subcellular structure of oilseeds.

3191. ROLE OF COTTONSEED IN THE FOOD FOR FREEDOM PROGRAM. Altschul, A. M. Cotton Gin & Oil Mill Press 67(13): 7-8. 1966.

The cottonseed industry, through its association and through the efforts of individual members and public research, has revolutionized the nature and the conception of its protein concentrate.

What was once considered suitable only for cattle feeding is now fed to nonruminant animals and to humans. It is a remarkable achievement. The cottonseed industry now has the opportunity to undertake activity toward offering edible cottonseed protein concentrate for inclusion in human feeding programs.

2948. OILSEED PROTEIN CONCENTRATES AS HUMAN FOOD. Altschul, A. M. J. Japanese Soc. Food Sci. Technol. 11: 400-406. 1964.

In view of the world shortage of protein supply, it is necessary to consider more seriously the role of oilseeds as a source of protein concentrates. This is not a new subject in Japan, where a highly developed technology exists for maximum utilization of the soybean. The virtue of oilseeds is that protein is concentrated by a relatively simple process of mechanical or solvent removal of the lipids. Protein concentrates prepared in this manner from soybeans, cottonseed, and groundnuts (peanuts) are described and their possibilities for incorporation in human diets considered. The limitations of these concentrates are discussed and means for improving their nutritional value and acceptability, as well as their structural properties, considered. The production of seed protein concentrates that will be nutritionally and aesthetically equivalent to animal protein will require a high degree of sophistication in the food industries. More information will be needed about the seed proteins themselves. Even those countries that now have enough animal protein can benefit from being able to include seed protein concentrates in their diet.

2622. RESEARCH CONTRIBUTIONS TO THE COTTONSEED INDUSTRY BY THE SOUTHERN REGIONAL RESEARCH LABORATORY. Lambou, M. G.; and Decossas, K. M. Cotton Gin & Oil Mill Press 64(22): 9-10, 27-32. 1963.

The SURDD has cooperated with the cottonseed industry and with other Government agencies and universities in research important to the industry. Selected achievements cited are those that have been commercialized, and an effort has been made to estimate the dollar value. These include: improved recovery of solvent and oil and improved color of oil from solvent-extracted cottonseed, improved preparation of cottonseed for processing, high-shear refining to improve color of cottonseed oils, filtration-extraction of cottonseed, a continuous feeder for the filtration-extraction process, and mechanical upgrading of cottonseed. Others have been discussed under "new methods" and include: a bench-scale method for evaluating the processing characteristics of oilseeds for filtration-extraction, methods for determining gossypol, the SURDD hardness tester, an improved method of analysis for available lysine, a method for determining cyclopropenoid acids, and an improved method for measuring oil color. Several new products are discussed, including the acetoglycerides, a high-quality cottonseed meal for meat birds and swine feeding, methyl esters from cottonseed soapstock, and improved cotton linters for use in papermaking. Dollar values of research developments are difficult to measure accurately. Our best estimates lead us to believe that cumulatively the cited achievements probably exceed the $35 million mark. However, it is believed that value of the indirect contributions of the SIRDD is basic, and exploratory research for which dollar values cannot presently be calculated exceeds the cumulative value of the more direct contributions. It is estimated that values of the direct and indirect contributions lie conservatively between $35 and $70 million.

2504. KING COTTON'S VALUABLE SEED. RECENT UTILIZATION OF COTTONSEED OIL AND MEAL. Decossas, K. M.; Weber, C. L.; and Patton, E. L. J. Am. Oil Chemists' Soc. 40: 4, 6-7, 16, 18. 1963.

Farmers receive approximately $250 million for the almost 6 million tons of cottonseed produced annually in the United States. More than 90 percent of this cottonseed is processed into its primary products--oil, meal, linters and hulls, and the 1.8 billion pounds of crude cottonseed oil and 2.5 million tons of cottonseed meal produced are sold by oil millers for more than $220 million and $140 million, respectively. About 80 percent of the cottonseed is processed in the South and the rest in California and Arizona. At the consumer level, cottonseed

products are valued at six times the farm value of cottonseed, or approximately $1.5 billion. Cottonseed oil accounted for 12 percent of total 1960 U.S. factory consumption of almost 10 billion pounds of fats and oils. Of the cottonseed oil consumed, 57 percent was used in salad or cooking oil, 30 percent in baking or frying fats, and 11 percent in margarine. Practically all of the 2.5 million tons of cottonseed meal produced was used for feed. It is estimated that 1.1 million tons of cottonseed meal were used by feed manufacturers in the preparation of mixed feeds during the 1960-61 season. Of this, 800,000 tons were used in cattle and sheep rations, 300,000 tons in poultry rations, and 20,000 tons in swine rations. Cottonseed meal prices were favorable during 1961, and consequently a considerable amount of cottonseed meal with gossypol content below .04 percent was used in swine supplements throughout the Middle West. Cottonseed flour sold for human consumption has averaged slightly over 3.5 million pounds annually since 1950.

2479. GOALS IN COTTONSEED PROCESSING. Hopper, T.H. Oil Mill Gaz. 67(8): 17-18. 1963.

Recent advances in the knowledge of composition, properties, and quality of cottonseed products and of nutrition lead to the belief that processors of cottonseed will become more concerned with constituents occurring in seed in trace or small amounts, such as gossypol, lysine, raffinose, cyclopropene acids, and tocopherols. To obtain the ideal meal, it appears necessary; (1) to reduce the total gossypol content to the lowest possible level, (2) to avoid disturbing the lysine of the seed protein, (3) to reduce the residual oil content to a minimum level, and (4) to eliminate the heat-labile antinutritional factor. Two approaches for improving oil quality are being investigated. One is to develop varieties producing glandless or gossypol-free cottonseed. The second approach is to find bleaching methods that will remove the alkali-insoluble red pigments resulting from the reaction of gossypol with oil constituents. There is every reason to believe that the full nutritive potential of cottonseed meal as a protein supplement for nonruminants can be attained.

2478. OLEAGINOUS MATERIALS FOR INDUSTRIAL USES. Fisher, C.H.; Lambou, M.G.; and Decossas, K.M. Oil Mill Gaz. 67(8): 7-11. 1963.

Cost of oleaginous materials is too high for extensive use in some of the largest markets, such as fuels and road building. However, they have the characteristics needed in raw materials for many major markets, including food products and various chemicals, chemical intermediates, polymers, protective coatings, and specialty products. Competing raw materials, particularly petroleum, natural gas, and coal tar, have profited from extremely low-cost and strong supporting research programs. Largely for this reason, these competing materials have captured some of the industrial outlets formerly dominated by oleaginous materials. However, intensified research on oils and fats in recent years has led to valuable achievements in the development of new food and nonfood products. Both economically and technologically, oils and fats can be expected to respond favorably to future research. It is believed that they will become increasingly important as raw materials for the manufacture of new and improved end-use products. The word "oleochemicals" has been suggested as a generic term to describe chemicals from oleaginous materials. There should be excellent opportunities worldwide for increasing both the food and nonfood utilization of glycerides, oils, and fats as a result of population increases and rising living standards.

2346. SEED PROTEINS AND WORLD FOOD PROBLEMS. Altschul, A.M. Econ. Botany 16: 2-13. 1962.

The problem of providing adequate protein for an expanding world population is discussed, with particular emphasis on the potential for obtaining added protein for humans from seeds. In order to understand the role plant proteins must play in increasing the protein supply, it is necessary to start with the present practice and analyze its benefits and shortcomings. Facets of the problem considered are the development of new methods, degree of sophistication necessary in processing these seeds, and new knowledge about seed proteins that must

be acquired. Examples drawn from the so-called oilseeds illustrate the discussion of these problems.

2336. CROPS AND THE CHEMICAL INDUSTRY. Fisher, C. H.; and Brunn, J. H. Yearbook Agr., U. S. Dept. Agr. 1962: 445-49.

More than a billion dollars' worth of farm products are sold each year to the chemical industry, whose production totals about 40 billion pounds a year. The article briefly describes some products and processes--important to the chemical and related industries--developed by the U. S. Department of Agriculture's utilization research laboratories. Emphasis is placed on products from vegetable oils, animal fats, naval stores, grains, starches, sugar, and molasses. The great potential for developing much larger markets for agricultural commodities through utilization research is discussed.

2321. RESEARCH ON COTTONSEED CONDUCTED THROUGH SRRL PROBABLY WILL BENEFIT ALL SEGMENTS OF CRUSHERS. Hopper, T. H. Cotton Trade J. 42(18): 13. May 4, 1962.

The U. S. Dept. of Agriculture is conducting research on improvement of the quality and properties of cottonseed meal and oil, on development of new-type specialty fats for the food industry, and on nonfood uses for oil. The National Cottonseed Products Association and the National Confectioners Association maintain fellowships in areas of their particular interest. Major effort has been on the improvement of nutritive value of cottonseed meal for feeding to poultry and swine. To obtain the greatest nutritive value and feeding response, it is necessary to remove gossypol, reduce the residual lipids to less than 1 percent, and to keep the lysine in available form. A meal approaching the ideal can be obtained by extraction of the oil with a mixture of acetone, hexane, and water. Engineering research is being conducted to develop a practical process. Alkali-fast pigments in badly off-colored cottonseed oils are being fractionated and characterized, and an alumina bleaching process that should aid in their removal is under investigation. Research on specialty fats has been concerned largely with studies on hydrogenation, the development of a cocoa butter-like fat, and means of making polyester-type fats. A simple process has been developed for the preparation of specific triglycerides without interesterification. A rapid procedure has been developed for converting solid-type triglycerides to their stable polymorphic form by mechanical working of the fat. New information has been obtained on the activity of Raney nickel catalyst that has an important bearing on the isomerization of fatty acids through hydrogenation. It has been found that morpholides of the selectively hydrogenated cottonseed fatty acids are useful as secondary plasticizers for vinyl-type plastics.

2218. FOR BETTER COTTONSEED OIL PRODUCTS. Hopper, T. H. Cotton Gin & Oil Mill Press 62(21): 22, 30. 1961.

Recent research on food uses of cottonseed oil is reviewed, with emphasis on acetoglycerides, confectioners' fats, new-type fat products, and on the development of new knowledge on the hydrogenation of oleic and linoleic acids. In addition, research on peanuts is reviewed. The need for research to improve present products and develop new ones from cottonseed oil is stressed as a means of cottonseed oil holding its premium position.

2192. PRESENT STATUS OF PROTEINS FROM OILSEEDS. Altschul, A. M. "Progress in Meeting Protein Needs of Infants and Pre-school Children, Proceedings of an International Conference, Washington, D. C., Aug. 21-24, 1960." Natl. Acad. Sci.--Natl. Research Council Publ. 843: 517-30. 1961.

Principles and practices involved in obtaining plant protein mixtures of high nutritive value are discussed. Subjects covered are the quantity of oilseeds potentially available for incorporation into mixtures suitable for human foods, problems to be solved in order that these materials can be used effectively in human diets, opportunities already existing for incorporating these materials into high-protein diets, and what can be expected as knowledge and technology advance.

2120. RESEARCH, A BASIS FOR CHANGE. Hopper, T. H. Cotton Gin & Oil Mill Press 62(10): 9, 32-33. 1961.

Recent progress at the SURDD on basic and technological research pertaining to the processing of cottonseed oil and cottonseed is reviewed. Brief descriptions are given of: (1) the use of activated alumina for bleaching off-colored cottonseed oil, (2) the production of meals of high nutritive quality by extraction of cottonseed with a mixture of acetone, hexane, and water, (3) the development of basic information and methods of analysis, and (4) the use of a textured belt for the cleaning of cottonseed.

2098. HOW AGRICULTURAL RESEARCH BENEFITS YOUR BUSINESS. Stansbury, M. F.; and Dawson, P. R. Mfg. Confectioner 41(3): 25-29. 1961.

The confectionery industry is one of our important food industries. Confections are reportedly distributed through more retail outlets than any other type of food. The total annual value of retail sales of confectionery products in the United States exceeds $2 billion, more than the amount spent for either soft drinks or ice cream. In past years the making of confectionery products has been an art to a greater extent than most other modern food processing operations. Within the past several decades, however, art is being replaced by scientific principles and technology. Agricultural research has played a role in this necessary change, and increased efficiency in agriculture has provided abundant quantities of raw materials for the confectionery industry at low cost. This report from the SURDD describes some of the research development of the four Utilization Research and Development Divisions of the U. S. Department of Agriculture that have been either of direct benefit to the confectionery industry or offer promise for applications in the future. Research needs of the industry are also discussed.

2082. NEW OPPORTUNITIES FOR SOUTHERN INDUSTRIES. Patton, E. L.; and Koltun, S. P. Southern Assoc. Sci. Ind. Feb. 1961. 10 pp. (Republished as "New Industrial Possibilities for Southern Agricultural Products," Atlanta Econ. Rev. 11(5): 17-20. 1961; and "New Industrial Possibilities for Middle South Agriculture Products," Middle South News, Winter 1961-1962: 12-13, 25-27.)

These selected examples of research and development activities of the SURDD emphasize that there are opportunities in the South for new industries based on southern-grown agricultural commodities. They emphasize too the growing role that can be and is being played by industry in establishing industrial plants to utilize surplus agricultural raw materials as efficiently and with the same quality and profit levels as comparable installations elsewhere in the United States.

2048. EQUIPMENT COSTS. Decossas, K. M.; Koltun, S. P.; and Patton, E. L. Chem. Eng. Progr. 56(12): 60-63. 1960.

Agricultural utilization research is offering industry numerous processes for producing new and improved products from cotton, oilseeds, citrus, pine gum, rice, and other farm commodities. These processes comprise a variety of unit operations, and have been evaluated over a wide range of productions based on both hypothetical and commercial operations. Equipment costs, largely obtained from equipment manufacturers during the past 6 years and used in the process evaluations, have been adjusted to the December 1959 level, classified for the various equipment units by material of construction and size, and charted, and are presented for quick reference and use.

2021. A REVIEW OF COST ANALYSIS IN AGRICULTURAL UTILIZATION RESEARCH AT THE SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION. Decossas, K. M.; Pollard, E. F.; and Patton, E. L. U. S. Dept. Agr. ARS 72-22, 16 pp. (Processed). 1960.

Cost research at the SURDD has been aimed at the evaluation of new and improved processes and products from utilization research on cotton, cottonseed, peanuts, tung, rice, fruits,

vegetables, sugarcane, and naval stores. Results of armchair cost estimates, preliminary cost estimates, and complete cost analyses are given. These results reveal that many of the new cotton chemical processes studied and developed at the Southern Regional Research Laboratory are inexpensive and show promise for commercial adoption. The cotton chemical processes evaluated include partial acetylation, full acetylation, aminization, partial carboxymethylation, carbamoylethylation, the THPC-methylolmelamine, APO-THPC, APO, and formic acid colloid of methylolmelamine resin treatments, hydroxyethylation, phosphonomethylation, and wash-wear. Costs calculated for producing gossypol and cocoa butter-like fat from cottonseed oil, and tung oil-resin varnish vehicle are also discussed.

2011. SURDD RESEARCH RELATED TO COTTONSEED PROCESSING. Hopper, T. H. Oil Mill Gaz. 65(4): 14-15. 1960.

A brief report on recent research related to cottonseed processing is given, including a description of a color index method for measurement of cottonseed oil color; the use of activated alumina for bleaching off-colored oils; the extraction of cottonseed with a solvent mixture consisting of acetone, hexane, and water to avoid loss of available lysine in the meals; and the SURDD belt cleaner for cleaning cottonseed.

2010. RESEARCH BY USDA ON NONFOOD USES OF FATS AND OILS. Hopper, T. H. Cotton Gin & Oil Mill Press 61(20): 14, 17. 1960.

The background of the need for research on the nonfood uses of fats and oils and their derived products is briefly reviewed, with citation of the research conducted by each of the four Utilization Research and Development Divisions by developing new uses in the manufacture of protective coatings, plastics, and detergents. Mention is made of improvements in the nutritive quality of oilseed meals and the use of fats in animal rations.

1862. CURRENT UTILIZATION OF COTTONSEED OIL AND MEAL. Decossas, K. M.; Pollard, E. F.; and Patton, E. L. Oil Mill Gaz. 64(4): 7-8, 10-11, 13-15. 1959.

Through the 1957-58 season, increasing amounts of cottonseed oil were consumed in the production of cooking oils, salad oils, mayonnaise, and related products, but total factory consumption of cottonseed oil decreased because decreasing amounts were used in shortening and margarine. Increasing amounts of cottonseed oil foots are being used as a feed additive. New uses are being found for cottonseed oil and foots. Cottonseed meal utilization in poultry and swine feeds has increased in the Southwest and West and decreased in the East, with the exception of the extreme Southeast. This geographical redistribution has resulted largely from price competition of soybean meal. Continuing research in gossypol chemistry and technology promises to improve the competitive position of cottonseed meal in these uses.

1756. RESEARCH ON PROTEINS--WHAT IT HAS MEANT TO US. Altschul, A. M. Cotton Gin & Oil Mill Press 60(10): 37, 86-87. 1959.

A brief history is given of the recognition of proteins as important nutritional materials and the progress in determining their composition and structure. One of the milestones has been the development of information on the amino-acid requirements of man. By relating this to the amino-acid composition of proteins, one can assess the relative value of various proteins for supplying the needed amino acids. As a result of the research on proteins, we are in a position to make more sophisticated use of proteins to the best advantage of a growing world population to the end that there will be adequate food for all and that each will get the kind of food that is best suited for good health and longevity. Further progress in this area depends on the progress of research on proteins.

1640. RESEARCH--A VITAL LINK BETWEEN AGRICULTURE AND INDUSTRY. Persell, R. M.; and Goheen, G. E. Proc. Louisiana Ind. Develop. Conf. 18-22. 1958.

Research, its inception, aims, and growth, forms the theme of this informal discussion. An infant industry, research has grown to $10 billion in less than 50 years; agriculture, $5 billion;

primary metals, $26 billion; petroleum, $20 billion; food processing, $47 billion; chemicals, $27 billion; and research, $10 billion. The human side of this 20th century miracle is presented--what research means to industry, to the farmer, and to the number of persons supported by one farmer (which has increased from about 12 persons in 1850 to 17 or 18 at present). The purposes of the overall agricultural research program of U. S. Department of Agriculture are outlined and utilization research is explained, with emphasis on the program of the SURDD.

1629. VEGETABLE OILS, FATS, AND WAXES. Dollear, F. G. Ind. Eng. Chem. 50: 48A-51A. 1958.

Progress in processing technology and chemistry of vegetable oils, fats, and waxes over the past 50 years is reviewed briefly. Hydrogenation to convert liquid oils into plastic fats was the first major achievement of this era. The introduction of soybeans in the Middle West about 1922 brought a new oilseed to this country, producing an oil that has found great utility in both edible and industrial products. Advances in vegetable oil processing have improved recovery and quality of oil, particularly the introduction of solvent-extraction processes. Lecithin and monoglycerides have entered the picture as food-grade emulsifiers. Ester interchange and directed interesterification have advanced from the laboratory to the commercial stage. The fat-soluble vitamins A, D, E, and K were discovered, and molecular distillation was introduced as a process for their recovery from vegetable oils. Protective coatings have undergone great changes. Primary change has been the introduction of alkyd resins and oil-modified alkyds having improved properties. The soapstock from refining of vegetable oils has been converted to fatty acids widely used in industrial products, among which are the detergents. Progress in wax technology has been primarily along the line of easier application and includes the development of self-polishing waxes. Improvements have been made in methods of chemical analysis for the determination of composition and the control of quality of fats and their derived products. More recent contributions have been in the fields of vapor-phase chromatographic separation and mass spectrometry. These new tools will make possible research to determine the influence of dietary fats on blood lipids and on health. Some predictions are made as to development that may occur over the next 5 or 10 years.

1620. OPPORTUNITIES IN THE FUTURE FOR COTTONSEED RESEARCH. Altschul, A. M. Cotton Gin & Oil Mill Press 59(3): 16, 33-35. 1958.

This is an attempt to project the future needs for research on cottonseed. New lines of research might be classified as: (1) those that develop from present research, (2) those that are results of general developments which may affect cottonseed, and (3) basic research. The major research at present is dominated by the existence of gossypol in the seed and the need to eliminate its deleterious effect. It is conceived that these problems will be solved, some by chemistry and some by breeding. When gossypol is out of the seed, there will be a need to explore for the presence of other materials that affect the value of cottonseed meal and oil in feeds and foods. There are several trends that might affect the cottonseed industry. These include the increased popularity of commercial foods and the need for new edible products from cottonseed oil, the increasing interchangeability of food products, the increased need for cheaper proteins for animal and man, and the increased concern over the relationship between diet and health. There will also be the need for basic research that seeks information to broaden our horizons and broaden our base of knowledge.

1542. RESEARCH ON IMPROVEMENT AND QUALITY OF COTTONSEED MEAL AND OIL. Altschul, A. M.; and Bensabat, L. S. Oil Mill Gaz. 62(4): 9-12. 1957.

Cottonseed meal is being used in increasing amounts in feeds for poultry and swine. Processing methods have been developed whereby meals as low as 0. 04 percent in free gossypol can be prepared. The methods producing the most nutritious meals are those using the minimum amount of heat. By measuring the lysine content of the meals it is possible to predict their nutritional performance in broiler rations. Strides have been made in

determining the chemical materials responsible for discoloration of eggs laid by hens fed cottonseed meals. In cottonseed oil research, effort has been made to determine the mechanism for color development; moreover, a procedure has been developed whereby poor quality cottonseed oils can be chemically treated to yield a final bleached oil of prime color.

1537. OILSEED RESEARCH PROGRESS. Hopper, T. H. Cotton Gin & Oil Mill Press 58(23): 30-33. 1957.

The progress and significance of recent oilseed research conducted at the SURDD is reviewed briefly, with attention called especially to research on (1) improving the nutritive value of cottonseed meal, (2) edible oil products, (3) industrial oil products, (4) oilseed processing, (5) soapstocks, (6) cleaning cottonseed, and (7) physical and chemical properties of constituents and components of oilseeds.

1532. IMPROVED COTTONSEED MEALS FOR FEEDING POULTRY AND SWINE, 1944-1955, AN ANNOTATED BIBLIOGRAPHY. Rubins, R. K.; Baringer, K. L.; and Skau, D. B. U. S. Dept. Agr. ARS-72-10, 91 pp. 1957. (Processed)

An abstract bibliography has been prepared on cottonseed meal for feeding to poultry and swine. Describing a cooperative project of State and Federal agencies and industry, the listing contains 159 titles, divided into six sections. Subjects include the program, its objectives and scope; pigments, their isolation and identification; effect of storage conditions, reaction of pigments, oral toxicity, and detoxification; gossypol; processing, effect on meal; processing, effect on protein; and feeds and feeding for poultry and swine. Cross-references are given at the end of each section.

1530. FAT PRODUCTS RESEARCH. NEW, SPECIAL FATS FOR CONFECTIONERY USE. Feuge, R. O. Candy Ind. Confectioners J. 109(10): 21, 42-43. 53. 1957.

Several developments concerned with the preparation and characterization of edible fats, particularly confectionery fats, have been selected from published articles of the SURDD and are described. Using data obtained in the characterization of acetoglycerides, it is shown how dilatometry, a field in which the Laboratory has contributed significantly, provides a number of items of important information, including coefficients of expansion, melting dilations, polymorphic transformations, and volume changes accompanying polymorphic transformations. A new instrument and technique developed for measuring the hardness of fats and waxes is described and discussed. Among new products developed at the Laboratory, the following are described: cocoa butter-like fats made from domestic oils, acetoglyceride products and their unique properties, and new-type polymeric fats made from domestic fats and short-chain dibasic acids.

1527. COTTONSEED. Hopper, T. H. Encyclopedia Britannica 6: 583-85. 1957.

For each pound of fiber, cotton plants yield approximately 2 pounds of seed. The world production of cottonseed in 1954 was over 20 million tons. Brazil, China, Egypt, India, Mexico, and the United States provide over one-half of the world supply. In 1954 the United States produced 5.7 million tons. The commercial importance of cottonseed is described, and the four principal products obtained through processing--oil, cake or meal, linters and hulls--are discussed.

1394. A REVIEW OF RESEARCH OF THE SOUTHERN BRANCH ON OILSEEDS. Goheen, G. E.; and Persell, R. M. Oil Mill Gaz. 61(3): 30-32. 1956.

Utilization research has as its general objective the discovery and development of new and improved methods to use farm crops. The agricultural utilization research program of the Department of Agriculture is outlined, and some of its accomplishments are cited. The oilseed program of the Southern Utilization Research Branch includes research to determine the composition of oilseeds and to acquire other important fundamental information, and to develop

new or improved processing methods and new or improved products. Among accomplishments in oilseed research at the Southern Utilization Research Branch are the filtration-extraction process for extraction of oils, new knowledge which has extended the utilization of cottonseed meal, improved formulations to control gelation of tung oil, new products such as the aceto-glycerides, and information that promises to develop many new industrial uses for fats and oils as raw materials.

1386. SOME RECENT DEVELOPMENTS AT THE SOUTHERN UTILIZATION RESEARCH BRANCH RELATED TO OIL MILLING. Gastrock, E. A. Oil Mill Gaz. 61(2): 22-23. 1956.

Major developments at the Southern Utilization Research Branch in recent years of interest to the oil milling industry are discussed and described briefly. The ARS Differentiator is a device for cleaning cottonseed and separating it according to grade. The Differentiator operates on a principle new for this purpose, that of slingshot projection. Progress in the commercial use of the filtration-extraction process is reported; after 2 years of commercial operation in a plant at Greenweed, Miss., early claims for the process based on bench-scale and pilot-plant developments have been equaled or bettered. Study of rates of extraction of raw and cooked cottonseed flakes, exhaustive extraction of cottonseed, application of filtration-extraction to minor oilseeds, and research related to the effect of processing on quality of products are discussed.

1380. NEW OPPORTUNITIES FOR COTTONSEED OIL AND MEAL THROUGH RESEARCH. Altschul, A. M. Cotton Gin & Oil Mill Press 57(14): 7-8, 45-47. 1956.

Research has shown that gossypol is one of the most important factors in determining quality of cottonseed meal and the color of cottonseed oil. Improved processing procedures are now needed to reach some of the following objectives: to keep gossypol from going into the oil and thus minimize the color problem, to eliminate the free-gossypol content of the meal, and thus make it suitable for unrestricted feeding to laying hens, and to produce meals of the highest possible protein quality. The paper is a general discussion of research on cottonseed products, past and present, and research needs.

1260. NEW WEALTH FROM AGRICULTURE THROUGH UTILIZATION RESEARCH. Fisher, C. H. Proc. South. Feed & Fert. Assoc. 1955-56.

The part played by research in modern life is touched briefly in the introduction, followed by a summary of the results of production research in agriculture. The greater portion of the paper is devoted to utilization research, a newcomer to the agricultural scene. Aims of agricultural utilization research are outlined, and some of the direct contributions by the four regional utilization research laboratories of the U. S. Department of Agriculture are given. Among the accomplishments described are the work on penicillin, development of rutin, improved cottonseed meal, the ARS filtration-extraction process, frozen orange concentrates, and a variety of developments from cotton research, such as the conforming cotton bandage, partly acetylated cotton, the cotton opener, tobacco shadecloth, and flame-resistant cotton fabrics. These and other research contributions are responsible for great benefits in improved living standards.

1224. NEW DEVELOPMENTS AND CURRENT PROBLEMS CONFRONTING COTTONSEED OIL MILLS. Gastrock, E. A.; and Vix, H. L. E. Oil Mill Gaz. 60(3): 19-21. 1955.

The authors discuss various phases of cottonseed oil mill operation from a practical point of view. After the processing method has been chosen from several now in general use, the operator must make a wise combination choice among several objectives, which may be grouped as operating efficiency and quality of product. The desirable features of a good process are outlined, and qualities of oil and meal from those in common use are compared. Major quality problems are enumerated. Industry and oilseed crop trends are also treated. In conclusion, cottonseed research underway at the Southern Utilization Research Branch is outlined.

1221. INFLUENCE OF COMMERCIAL PROCESSING CONDITIONS ON PROPERTIES OF COTTONSEED PRODUCTS. Hopper, T. H. Oil Mill Gaz. 60(3): 14, 16-17. 1955.

The influence of conditions imposed by the several commercial methods of processing cottonseed oil and meal are reviewed briefly. The variables discussed are composition of kernels and meal, properties of oil, and processing methods. Comparative data on the influence of types of processing methods on the free-gossypol content and nitrogen solubility of some cottonseed meals are summarized.

1164. RESEARCH ON COTTONSEED MEALS. Jensen, E. A. Cotton Gin & Oil Mill Press 56(12): 50-53. 1955.

Progress is reported on a chemical method for estimating the nutritive value of cottonseed meal. Work has been directed toward determination of the amino-acids content and the composition of isolated protein fractions. A technique of paper chromatography was modified to obtain an excellent two-dimensional chromatogram of a cottonseed hydrolyzate. For the protein investigations, a fractionation procedure was applied to two meals; one, a standard meal of high nutritive value; the other, a portion of the standard that has been autoclaved until its nutritive value is reduced approximately 60 percent. Water extraction removed approximately the same weight of material from each meal. Reduction in the nutritive value of the autoclaved meal was not reflected in a change in its water-soluble nitrogenous constituents. When fractionated with 0. 02 N sodium chloride solution, 28 percent of the residue from the unautoclaved sample was salt soluble, and only 2 percent from the autoclaved sample. The salt-soluble material from the unautoclaved sample contained approximately 17 percent nitrogen, or 30 percent of the original nitrogen, and that from the autoclaved sample contained only 5 percent nitrogen, or less than 1 percent of the original nitrogen.

1110. OILSEED RESEARCH AT SRRL: A PROGRESS REPORT. Kime, J. A. Oil Mill Gaz. 59(2): 34-37. 1954.

The oilseed research program at Southern Regional Research Laboratory is reviewed in some detail, the various lines of investigation are outlined, and accomplishments are discussed. The major emphasis in oilseed research is on cottonseed, but utilization of peanuts, tung, and minor oil-bearing substances, such as sesame and rice bran, are also investigated.

1086. COTTONSEED. Hopper, T. H. Encyclopedia Britannica, Vol. 6: pages 583-585. Encyclopedia Britannica, Inc., Chicago, Illinois. 1954. (Revision of article by D. F. J. Lynch, Encyclopedia Britannica, Vol. 6: pages 584-586. 1944.)

This article traces the development of the use of cottonseed as a source of oil, meal, linters, and hulls. This development parallels almost wholly a history of the development of the cottonseed industry in the United States. The processing of cottonseed to recover products and the use of these products are briefly described. Recent statistics are given on the production and value of cottonseed and cottonseed products for the United States.

1035. FOOD PROCESSING RESEARCH ON SOUTHERN FARM CROPS. Fisher, C. H.; and Persell, R. M. South. Food Processor (Ann. Rev. No.) 1954.

Progress is being made in solving some important processing problems of the South through research on citrus fruits, cucumbers, sweet potatoes, beets, tomatoes, blackeyed peas, rice, cottonseed, peanuts, and sugarcane.

1034. THE NEED FOR AGRICULTURAL RESEARCH IN THE SOUTH AND SOUTHWEST. Fisher, C. H.; and Persell, R. M. Cotton Gin & Oil Mill Press 55(8): 11-12, 48-52. 1954.

Research will be the basis for any sound, long-range program for agriculture, and will be keynoted by cooperation among universities, industry, government research groups, and trade and

commodity associations. Expectations from research, the rapid growth of industrial research as a major influence in industrial development, the story of agricultural research, the revolutionary progress of agriculture in the last 75 years in the United States, the effect of research in creating need for greater knowledge to solve new problems, the need for "balance" in agricultural research, the problem of agricultural surpluses; this country's need to adjust to worldwide changes in economic and social conditions, and research to discover entirely novel operations are main topics discussed.

820. BETTER PRODUCTS FROM COTTONSEED THROUGH RESEARCH, AIM OF PROJECT. Altschul, A. M.; and Baringer, K. L. Cotton Trade J. 34(6): 50-52. 1952.

Aims and results of a cooperative research program between the Southern Regional Research Laboratory and other Federal agencies, State experiment stations, the National Cottonseed Products Association, and cottonseed oil mills, whose objective is to improve the nutritive value of cottonseed meal without impairing oil quality, are described.

776. UTILIZATION OF THE BYPRODUCTS OF SOUTHERN AGRICULTURE. Fisher, C. H. J. South. Research 4(2): 16-19. 1952.

Progress in developing uses for agricultural byproducts of the South as a means of overcoming problems associated with the disposal of residues and so-called waste materials is summarized, with examples drawn principally from the results of research at the four regional utilization laboratories of the U. S. Department of Agriculture.

775. FOOD PROCESSING--A FIELD OF USDA RESEARCH ON CROP UTILIZATION IN THE SOUTH. Fisher, C. H.; and Crow, J. H. South. Food Processor (Ann. Rev. No.) 1952.

Highlights in studies in 1951 on citrus fruits, cucumbers, sugarcane, peanut butter, and vegetable oils conducted at the Southern Regional Research Laboratory and at its field stations are reported.

756. VEGETABLE OILS' RISING PRESTIGE. Markley, K. S. Southwest Retort 4(4): 10-11. 1952.

The following crops account for about 90 percent of all the vegetable oils of commerce: coconut, peanuts, cottonseed, soybean, rape, mustard, linseed, olive, palm, sesame, sunflower, palm kernel, tung, and babassu. Production statistics are given for each of these crops. Research to realize the potentialities of sesame seed is mentioned.

650. SOME REMARKS ON OILSEEDS RESEARCH AT THE SOUTHERN REGIONAL RESEARCH LABORATORY. Fisher, C. H. Natl. Oil Mill Supt. Assoc. Proc., 57th Ann. Conv. 1951. Oil Mill Gaz. 56(1): 32-34. 1951.

Research on cottonseed and other oilseeds at the Southern Regional Research Laboratory and related research on oilseeds conducted in the other regional research laboratories are discussed. Examples given of cottonseed research include cooperative work on the meal and chemical studies of gossypol.

558. VEGETABLE OIL CROPS AS FOOD, FEED, AND INDUSTRIAL RAW MATERIALS. Markley, K. S. Cotton Gin & Oil Mill Press 52(11): 18-21, 47-53. 1951.

This article is a general review of world production and consumption of vegetable fats and oils, including source and types of oil-bearing materials, areas of production, yields per unit of area, oil and protein contents, and quality grades and uses. This information is given in detail for the principal oil crops: soybeans, cottonseed, peanuts, corn, olives, coconut, oil palm, flax, tung, and castor seed, which together account for nearly 90 percent of all the

vegetable oils of commerce. The average yield of all oil-bearing materials varies from 63 percent for copra to 16 percent for soybeans. The average yield of meal varies from 35 percent for copra to 80 percent for soybeans. The range of yields of oil per acre is from 2,500 pounds to the acre for the African oil palm to 70 pounds for cottonseed. In general, the higher the oil content of the seeds, the lower is the protein content, and vice versa. Oilseed meals contribute a great deal of the feed for livestock and, thus, indirectly, to fat supplies; and have uses also in food products and in several industrial products. Oilseeds are chemically very complex, their quality and condition being determined by variety, soil, weather during growth and ripening, cultural practices, and the treatment they receive after harvesting.

499. PROBLEMS AND PROGRESS IN THE SOLVENT EXTRACTION OF COTTONSEED. Persell, R. M.; Pollard, E. F.; and Gastrock, E. A. Cotton Gin & Oil Mill Press 51(26): 9-11, 24-25, 30-32. 1950.

The introduction of solvent extraction to the cottonseed industry has made possible a higher recovery of the oil and production of a meal that is highly nutritious for animal and human consumption, as well as being a potential source of industrial proteins. Many complex economic and technological problems must be solved before the full theoretical advantage of the process can be realized. Some of these problems are being worked out in commercial plants and others in research laboratories. The scope and importance of these problems in these times of rapid technological advances and increasingly competitive conditions make this a fertile field for research.

464. THE ADVANCING UTILIZATION OF SOUTHERN FARM CROPS. Kime, J. A. Texas Agricultural Workers' Assoc., Proc., 23rd Ann. Meeting, January 13-14, 1950, pp. 39-45. Also a paraphrase, Oil Mill Gaz. 55(1): 25-27. 1950. (Natl. Oil Mill Superintendents Assoc., Proc.)

Emphasizing the effectiveness of cooperation between the Southern Regional Research Laboratory and other agricultural workers in coping with practical problems of mutual concern, the paper cites numerous instances of successful joint research, in Texas particularly. The overall research program of the Southern Regional Research Laboratory covers practically every step of processing that could be expected to improve the industrial utilization of cotton, cottonseed, sweetpotatoes, rice, peanuts, and other oilseeds. A few major cooperative achievements are reviewed.

386. CHEMURGIC ADVANCES IN COTTON AND PEANUTS. Scott, W. M. Chemurgic Dig. 8(5): 26-8. 1949.

The relationship between chemurgy and the work of the U. S. Department of Agriculture Regional Research Laboratories is emphasized. Examples are cited of research in New Orleans that broadened the potential industrial future of cotton and peanuts. Although the overall program of the Southern Regional Research Laboratory is discussed, the paper essentially is a report on progress made during the past year in research aimed at improving the economic position of these two crops in relation to that of competing products.

312. RESEARCH AS AN AID TO COTTON AND COTTONSEED IN MAINTAINING MARKETS. Scott, W. M. U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-185. 8 pp. (Processed). 1948.

W. M. Scott identifies the aim of research in the U. S. Department of Agriculture's Regional Research Laboratories with the definition of chemurgy, then outlines the role of the Southern Regional Research Laboratory in increasing industrial utilization of cotton and cottonseed. The Southern Regional Research Laboratory has made definite strides in this direction by various developments cited.

290. THE RESEARCH FELLOWSHIP OF THE NATIONAL COTTONSEED PRODUCTS ASSOCIATION. Swift, C. E. J. Am. Oil Chemists' Soc. 24: 412-16. 1947.

A brief history is given of the origin, purposes, and accomplishments of the Research Fellowship of the National Cottonseed Products Association, including brief biographies of previous holders of the Fellowship.

223. THE COTTONSEED PROCESSING INDUSTRY. Markley, K. S. Cotton & Cotton Oil Press 47: A-3, A-6. 1946.

From 1870 until about 1915 the cottonseed milling industry developed phenomenally. Then the number of active mills declined, dropping from 885 during 1914-15, to 727 by 1918, and to about 360 in 1945-46. The volume of seed crushed has remained relatively constant during the last 30 years, with 4,491,000 tons crushed during the 10-year period 1909-18 by 813 mills, as compared to 4,253,000 tons crushed during 1944-45 by 382 mills. The seasonal nature of cottonseed processing has favored the hydraulic press as the most economical method of crushing . Nearly all disadvantages of the hydraulic press are overcome by the continuous screw-press method. Both methods result in incomplete recovery of the oil and considerable denaturation of the protein of the cake. During the 1930's, solvent extraction, which had originated earlier in Europe, was applied in the United States on a large scale to the recovery of oil from soybeans, and the success achieved has stimulated interest in use of the method for other oilseed, particularly cottonseed. It is difficult to apply direct solvent extraction to seeds of high oil content, and in Europe such oilseeds are usually forepressed in screw press before solvent extraction. In the United States attempts have been made to apply direct solvent extraction without prior forepressing. The Southern Regional Research Laboratory has developed on a laboratory and semi-pilot-plant scale a unique flotation method that separates cottonseed into three distinct components: intact pigment glands, an oil-solvent mixture, and a substantially oil-free, gland-free meal.

165. FAT AND OIL RESOURCES OF LATIN AMERICA. Markley, K. S. Plants and Plant Science in Latin America, ed. by Frans Verdoorn. Chronica Botanica Co., Waltham, Massachusetts, pp. 211-18. 1945.

The Latin American Republics are discussed as an actual and potential source of vegetable fats and oils, with emphasis upon the potentialities of production. With the application of modern agricultural methods in these countries, domestic needs could be supplied, as well as deficits elsewhere in the Western Hemisphere. Treated from the standpoint of individual plant sources and grouped as far as possible into wild, semicultivated, and cultivated sources, data are presented on production of the following plants: babassu; coconut and related palms; dende palm; oiticica and related oils, cocoa and ucuuba butters; castor beans; carnauba, uricuri, candelilla, and other waxes; linseed and tung oil; sunflower, cottonseed, and cottonseed oil; peanut oil; and a group of miscellaneous oils and oilseeds. Argentina, Brazil, and Mexico appear to be the countries having the greatest possibilities.

27. VEGETABLE OILS THAT THE UNITED STATES NEEDS FROM THE AMERICAN TROPICS. Markley, K. S. Oil, Paint & Drug Reptr. 142(4): 7, 51, 61; (6): 7, 53, 55; (7): 7;(8): 7, 47. 1942. U. S. Dept. Agr., Bur. Agr. Chem. and Eng. ACE-173, 27 pp. 1942. (Processed).

Consumption of fats and oils in the United States, amounts produced domestically, and the amounts, kind, and sources of those imported are discussed in considerable detail. End uses of imported fats and oils are described. The paper includes a brief bibliography, a table of statistics on oils and oil-bearing materials imported from Latin America, and tables of statistics on several of the most important oils and oil-bearing materials, including lauric acid-type soap oils, murumuru and tucum kernels, oiticica, castor beans, palm, and flaxseed. Tables on U. S. consumption of drying oils and imports of hard waxes are also included.

10. TECHNOLOGY OF THE COTTONSEED CRUSHING INDUSTRY. Markley, K. S.; and Lynch, D. F. J. Cotton Research Congr. Proc. 1: 211-24. 1940.

The development of the cottonseed industry to the present date is reviewed briefly. Various phases of processing technology now in use are discussed. Sections or topics include oil extraction, refinery operations, bleaching of the oil, winterization, deodorization, and hydrogenation.

9. THE COTTONSEED INDUSTRY AND THE SOUTHERN REGIONAL LABORATORY. Gastrock, E. A.; and Markley, K. S. Oil Mill Gaz. 44(12): 29-31. 1940. (Republished, Ibid. 45(1): 15-18, 20-21. 1940.)

Background of the establishment of the regional research laboratories is presented briefly, progress on erection of the building in New Orleans is reported, and plans for operation of the research program are outlined. The authors then discuss the history and importance of the cottonseed industry, with statistical background. Need for research on cottonseed and its three major products, oil, protein, and linters, their derivatives, and potential uses is then discussed in some detail.

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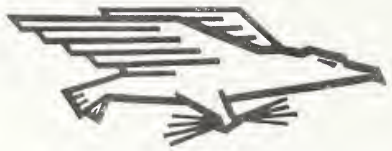

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